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The Journal of the SOCIETY OF DYERS AND COLOURISTS

Volume 67 Number 10

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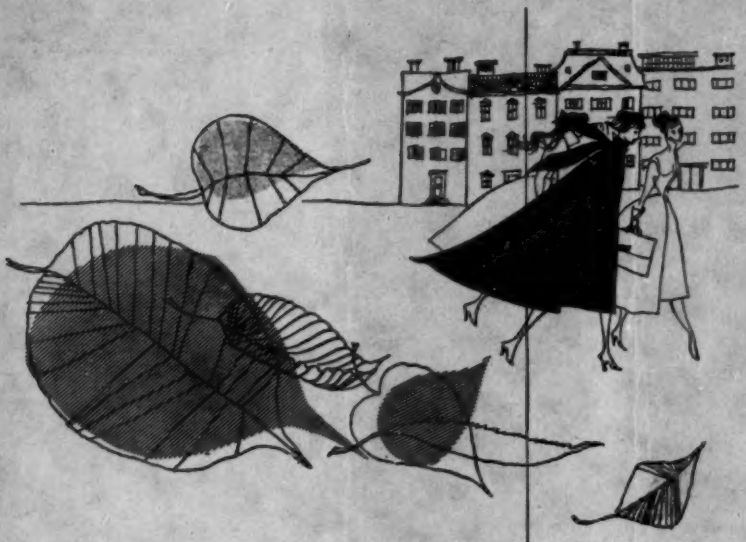

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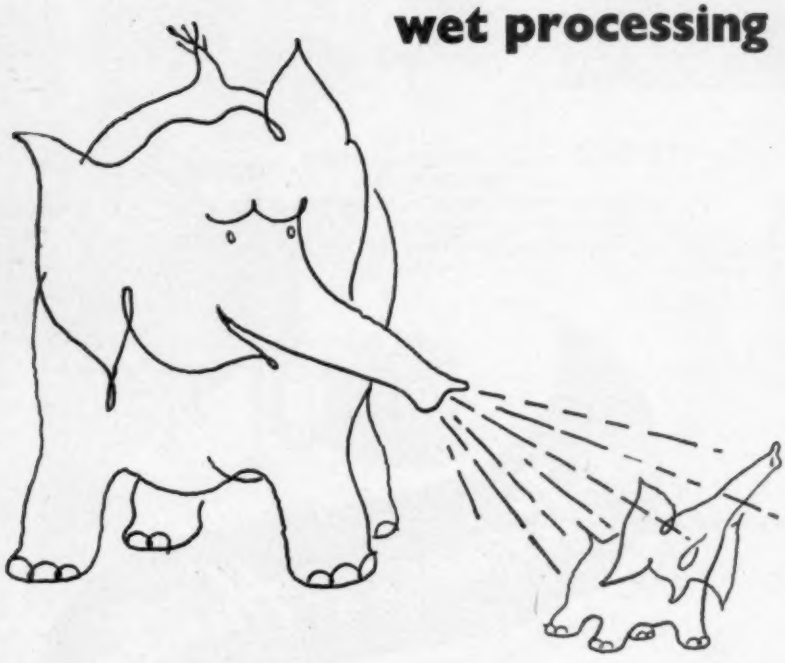

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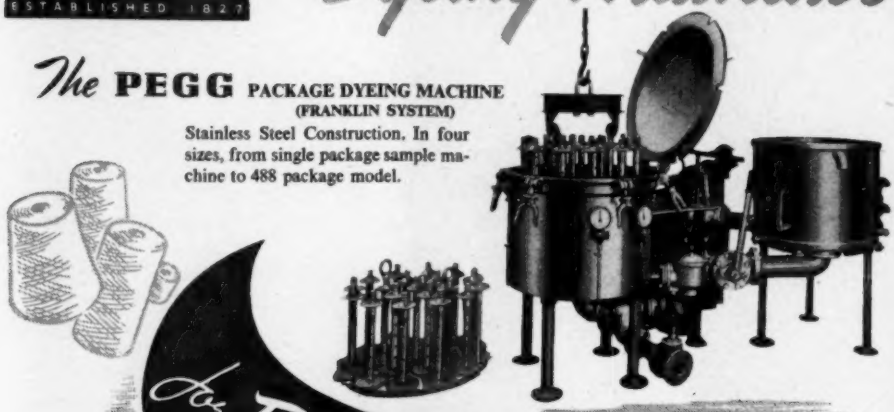

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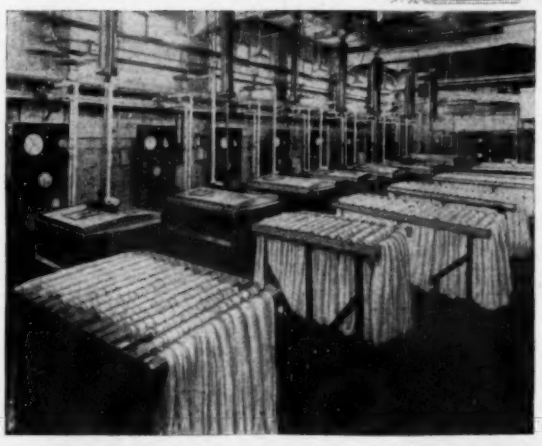

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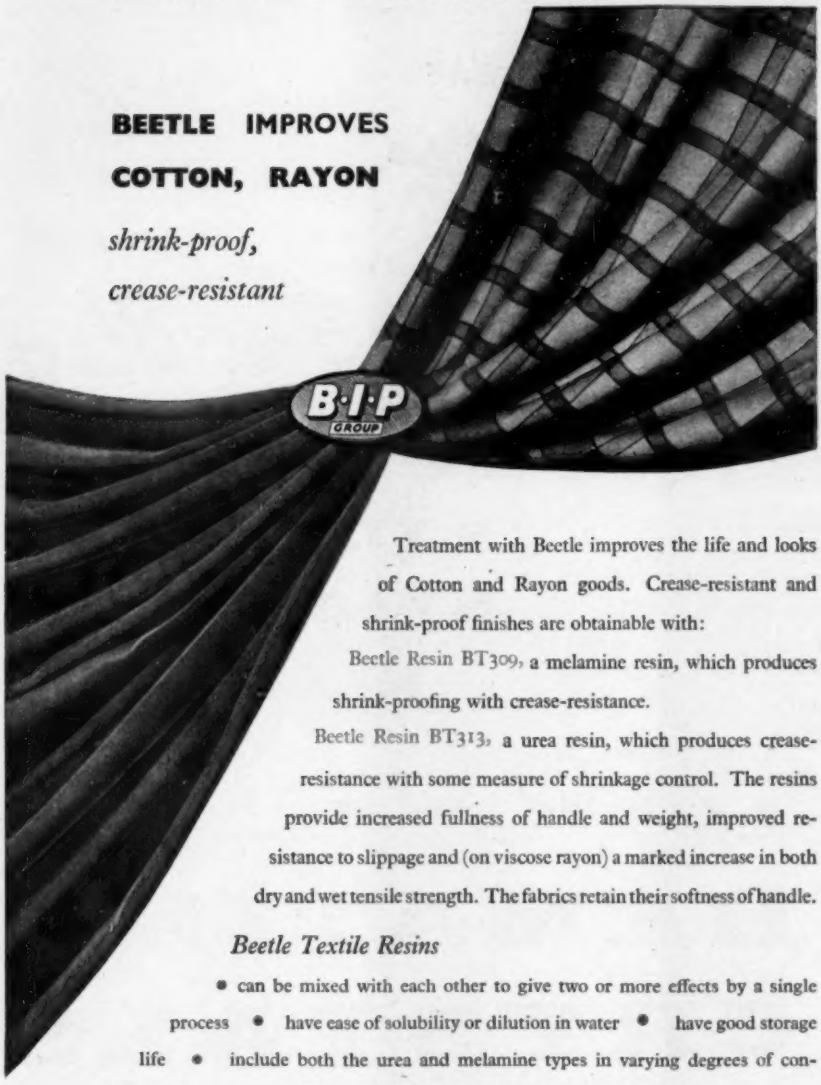

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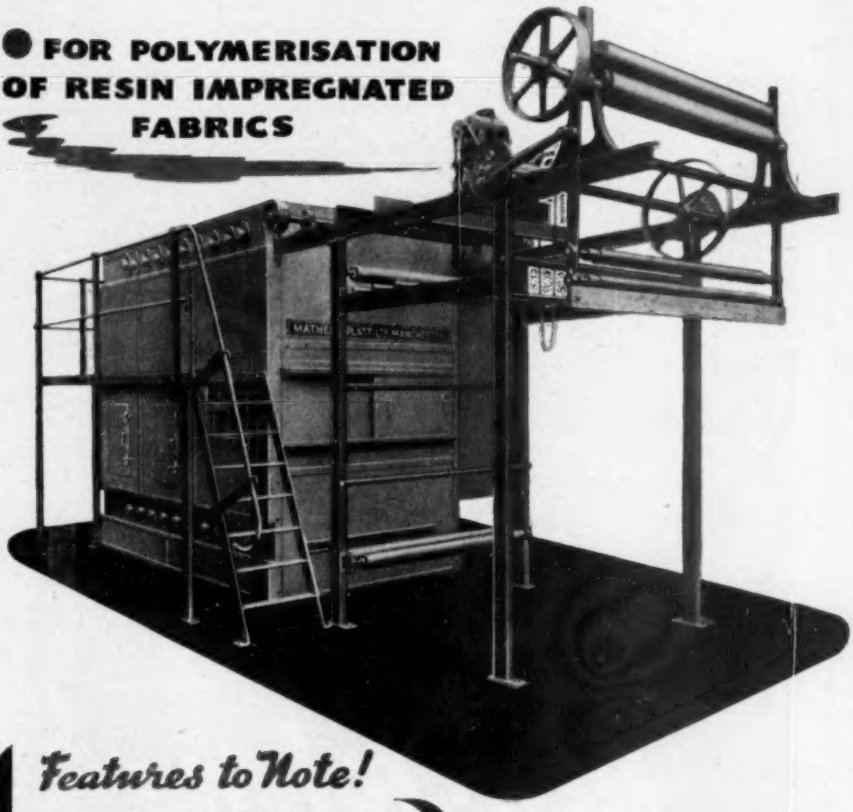

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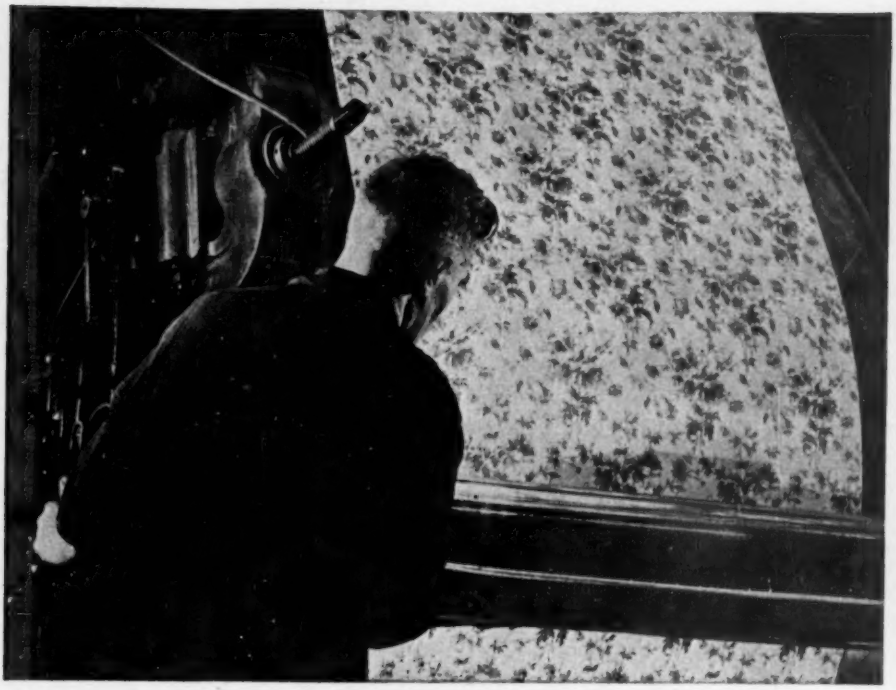

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NOTICE TO MEMBERS AND SUBSCRIBERS

Readers requiring *general information* regarding the Official Notices, List of Officers of the Society, etc. should consult pages 1–6 of the January 1951 and pages 253–256 of the July 1951 issues of the *Journal*, or write to *The General Secretary*, The Society of Dyers and Colourists, 32–34 Piccadilly, Bradford, Yorkshire (*Telephone* Bradford 25138–9). *Editorial Communications* should be addressed to *The Editor*, at the same address.

Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

LECTURES

Rayon Staple— A Dyeing Miscellany — *J. Boulton*

The pH Control of Acid and Chrome Dyeing—
II— Single-bath Mordant Dyeing — *G. H. Lister*

COMMUNICATIONS

The Colorimetric Determination of Indigo
J. Lotichius and J. Kooyman

Old Fulling Methods — *K. G. Ponting*

The Removal of Impurities from Grey Cotton—
I— A Review of Developments in Plant for Alkali Boiling
II— Experiments with an Alkali Pad–Steaming Sequence
H. A. Turner and O. J. Hvattum

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Forthcoming Meetings of the Society

1951
Thursday 1st Nov. — PERKIN MEDAL 1950—Professor J. B. Speakman, D.Sc. F.R.I.C., F.T.I. *The Structure and Reactivity of Keratin.* Great Northern Victoria Hotel, Bradford. 7.30 p.m.

Saturdays—20th October to 24th November 1951
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HUDDERSFIELD SECTION

All meetings held at Field's Café, Huddersfield at 7.30 p.m., unless otherwise stated

1951

Friday 2nd Nov. — SEVENTH ANNUAL DINNER Heywoods Cafe, Huddersfield.

Tuesday 20th Nov. — J. W. Reidy, Esq. (L. B. Holliday & Co. Ltd.) *Some Modern Trends in Dyeing Technique*

Tuesday 18th Dec. — Dr. G. T. Douglas (I.C.I. Ltd.) *The Dyeing of 'Ardil' Protein Fibre*

1952

Tuesday 15th Jan. — D. Hanson, Esq. *A Chemist in the Woollen Industry*

Tuesday 19th Feb. — F. Jordinson, Esq. *Defective Colour Vision*

Tuesday 18th March — Dr. C. B. Stevens *Some Observations on Dyeing Properties of Modified Wool*

Tuesday 8th April — ANNUAL GENERAL MEETING Dr. G. H. Lister (Sandoz Products Ltd.) *Acid and Chrome Dyes on Wool*

LONDON SECTION

All meetings held at 6 p.m. in the Rooms of the Royal Society, Burlington House, Piccadilly, London W.1, unless otherwise stated

1951

Friday 2nd Nov. — W. S. Wood, Esq. and Dr. K. W. Richmond (Laporte Chemicals Ltd.). *Recent Developments in Bleaching with Hydrogen Peroxide*

Friday 7th Dec. — G. S. J. White, Esq. (I.C.I.). *Recent Developments in Dyes and Colouring*

1952

Friday 4th Jan. — J. E. Ford, Esq. and W. J. Roff, Esq. (B.C.I.R.A.). *The Identification of Fibres, New and Old*

Friday 1st Feb. — N. Jackson, Esq. (Courtaulds). *Faults and Damages in Fabrics*

Friday 7th Mar. — Three short papers followed by a questionnaire on *Problems of the Garment Dyer and Cleaner*

It is also intended to hold a Dinner and Dance in the New Year, and to arrange for a lecture to be given in Luton in the spring, on a subject of interest to the hat industry. The date for the Annual General Meeting will be fixed later, for some time shortly after the last meeting of the session.

MANCHESTER SECTION

1951

Thursday 1st Nov. — (At the invitation of the Plastics Institute, North-Western Section). Professor W. T. Astbury. *Synthetic Fibres, Past and Present.* Manchester College of Technology

Friday 9th Nov. — A lecture will be held at Bury. The details of which will be announced later

Friday 23rd Nov. — LADIES' EVENING, *Man as a Spider*, details to be announced later. Manchester College of Technology. 7 p.m.

Continued on page xxix

Manchester Section—*continued*

Friday 21st Dec. — (Joint with the British Association of Managers of Textile Works), discussion on Dyeing Machinery, introduced by F. H. Marsh, Esq. (Longclose Engineering Co. Ltd.), Midland Hotel, Manchester. 7 p.m.

1952

Friday 18th Jan. — Details to be announced later, Gas Showrooms, Town Hall Extension, Manchester at 6.30 p.m.

Friday 15th Feb. — E. H. Waters, Esq. and H. Sumner, Esq. *The Effect of Soaping of Vat Dyes.* Gas Showrooms, Town Hall Extension, Manchester. 6.30 p.m.

Thursday 6th March — (Joint with the Textile Institute, Lancashire Section), Dr. A. Landolt (Ciba Ltd.) *Modern Developments in Textile Auxiliaries.* Textile Institute. 7 p.m.

Friday 21st March — Symposium on *Systems of Pack Dyeing.* Gas Showrooms, Town Hall Extension, Manchester. 6.30 p.m.

Friday 18th April — Annual General Meeting, details to be announced later. Gas Showrooms, Town Hall Extension, Manchester. 6.30 p.m.

MIDLANDS SECTION

All meetings commence at 7 p.m.

1951

Wednesday 31st Oct. — J. S. Ward, Esq., B.Sc. and H. Hampson, Esq. *Some Investigations into the Dyeing of Cellulose Acetate.* Victoria Station Hotel, Nottingham

Wednesday 21st Nov. — H. R. Hadfield, Esq., M.Sc. *The Levelling of Water-Soluble Dyes on Nylon.* College of Technology, Leicester

Friday 30th Nov. — LADIES' SOCIAL EVENING King's Head Hotel, Loughborough

Wednesday 12th Dec. — *Colloquium.* Short Papers on Practical Problems given by Local Members. Victoria Station Hotel, Nottingham

1952

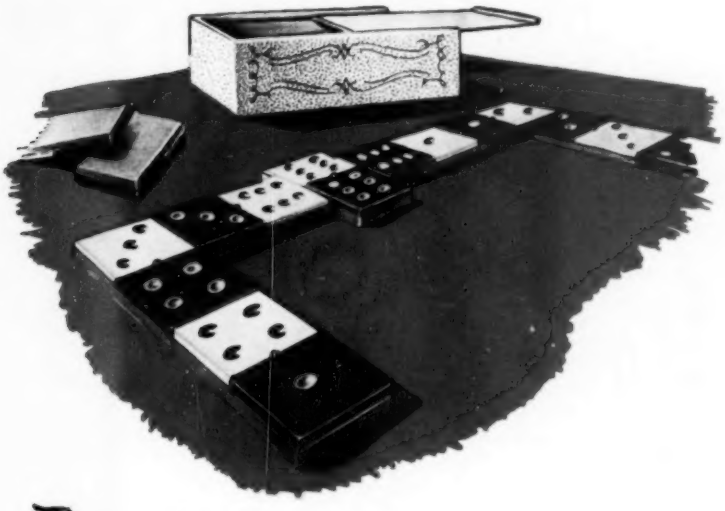
Friday 18th Jan. — Discussion Meeting with the Leicester Textile Society. *The Dyer's Limitations* (With Particular Reference to Mixed Fibres). College of Technology, Leicester

Friday 22nd Feb. — MIDLANDS SECTION DINNER Black Boy Hotel, Nottingham

Wednesday 27th Feb. — G. G. Taylor, Esq., B.Sc., A.Inst.P. *The Uses of Microscopy in Textile Dyeing and Finishing* (Joint Meeting with the British Association of Chemists). Midland Hotel, Derby

Wednesday 26th March — G. H. Lister, Esq., Ph.D., B.Sc. *The Influence of Dyeing Methods on Fibre Damage.* College of Technology, Leicester

Wednesday 23rd April — G. K. Mecklenburgh, Esq., S. Shaw, Esq., and H. W. Peters, Esq. *Some Developments in Setting and Finishing Nylon Textiles* (Preceded at 6.45 p.m. by the Annual General Meeting of the Section). King's Head Hotel, Loughborough



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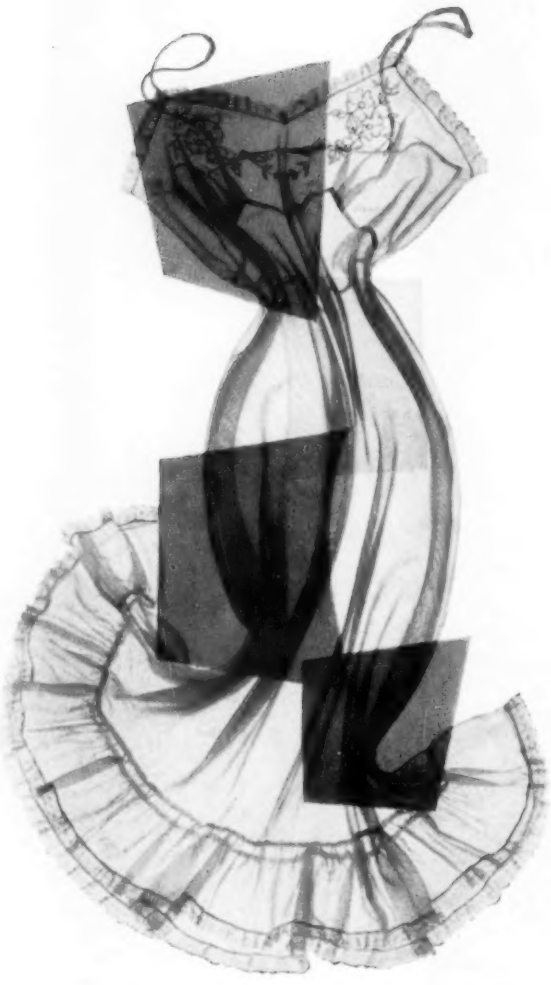

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THE JOURNAL

OF THE

Society of Dyers and Colourists

Volume 67—Number 10 OCTOBER 1951 *Issued Monthly*

Proceedings of the Society

Causes of Unlevel Dyeing of Cotton Cellulose

The Influence of Mercerising and Bleaching Processes on the Fine Structure of Cellulose

I. Gailey

Meeting of the Scottish Section held at the St. Enoch Hotel, Glasgow, on 20th February 1951, Mr. D. B. F. McAndrew in the chair

The worst types of unlevelness are caused by the presence of certain impurities on the surface of cotton. These impurities fall into three groups—(1) Substances that are inert and prevent normal contact between cellulose and dye. (2) Substances that are chemically active, and attract or repel dyes. (3) Substances that exert no effect.

Unlevelness of a less striking character can also be caused by differences in the sorptive capacity of the cellulose. Such differences in the sorptive capacity can be produced during mercerising, kier boiling, and drying.

Unlevelness in dyeing is a feature that is met only too frequently in the dyehouse, and it has been and is the custom to blame the dyer. It is hoped to show in this paper, however, that in many instances the dyer is merely suffering the consequences of faults that have arisen in preceding processes.

Work reported in this paper deals with causes of unlevelness arising from the condition of cotton, particularly bleached cotton, and does not deal with those causes that are initiated in the dyebath.

Variations in the cotton substrate causing unlevelness can be divided into four main classes—

(A) Presence of impurities in cotton. These impurities can be classified as follows—

(i) Substances which are inert and which act as a barrier obstructing normal contact between cellulose and dye, e.g. petroleum jellies, oils, mutton tallow, wax, etc.

(ii) Substances that are chemically active and that preferentially absorb or repel dyes, e.g. pectic matter, size.

(iii) Substances that exert no effect, e.g. cotton wax.

(B) Variations in sorptive capacity caused by chemical modifications of cellulose, e.g. oxycellulose and hydrocellulose. These modified forms of cellulose behave in a similar manner to substances listed above in A (ii).

(C) Optical effects.

(D) Variations in the sorptive capacity of cotton arising from differences in the fine structure of cellulose.

While some of the worst types of unlevelness are due to A–C, it is not proposed to deal with these in this paper, since they have been reported very fully elsewhere by others [1,2].

It is now generally accepted that the dyeing properties of cotton are determined by the availability of functional groups in the cellulose molecules. The functional groups in cellulose are hydroxyl groups, and the ones that undergo reaction are those that are present in the amorphous regions of the cellulose and on the surfaces of the crystalline portions. If, by any means, the proportion of available hydroxyl groups in one part of a material is altered, then unlevelness may result.

The rest of the paper is confined to considering those factors that alter the availability of hydroxyl groups in the macromolecular cellulose structure.

Variation in Sorptive Capacity of Cotton Cellulose Caused by Differences in Fine Structure

A good deal of work has been done in recent years to determine the effect of the fine structure of cellulose on the chemical and physical properties of cotton and viscose.

Many of the results obtained from work on viscose are directly applicable to cotton, since there

is much in common between the two substances. While there are many points of similarity, there are several points of difference between them, such as degree of polymerisation, percentage crystallinity, dimensions of crystallites, and composition of functional groups (viscose has a higher proportion of carboxyl groups). These differences result in differences in behaviour between cotton and viscose under certain conditions.

Contrary to older ideas, cellulose is fairly reactive and readily undergoes change. If a change takes place in an uncontrolled manner, irregular sorption is often the result.

Twenty years ago it was shown that reactivity of cellulose, as measured by the ease with which it is hydrolysed by acid, and the sorptive capacity of cellulose for moisture, dyes, and alkali are related [3]. Neale [4] concluded about the same time that the sorptive capacity of cellulose was determined by the availability of hydroxyl groups. In more recent years attempts have been made to relate sorptive capacity with the proportion of amorphous cellulose present in cotton, viscose, etc. P. H. Hermans has shown that, as a first approximation, there is a linear relationship between these quantities [5]. Support for this theory has appeared in a number of recent publications [6]. Experiments in which the percentage crystallinity was altered have confirmed the linear relationship between sorptive capacity and crystallinity [7,8].

The decrease in sorptive capacity with increased crystallinity has been explained as follows. The sorptive capacity of cellulose is a measure of the accessibility of hydroxyl groups. The hydroxyl groups throughout the amorphous regions, but only those on the surfaces of crystalline regions, are accessible. Hence any increase in crystallinity results in a reduction of hydroxyl groups available to undergo reaction.

Crystallisation is not the only process that brings about a reduction in number of available hydroxyl groups. Stretching during mercerising reduces the sorptive capacity of cellulose, as shown by Table I. Stretching of viscose during spinning has a similar effect [10,11].

TABLE I

Increase in Length of Thread after Mercerising (%)	Sorptive Capacity (Barium Activity No.*)
0	149
2·2	145
4·4	142

* The "barium activity number" is defined as—

$$\frac{\text{Wt. of } Ba(OH)_2 \text{ sorbed per gram of cellulose sample} \times 100}{\text{Wt. of } Ba(OH)_2 \text{ sorbed per gram of scoured grey unmercerised cotton}}$$

This method of measuring the sorptive capacity of cellulose was introduced by Neale [4], and Edelstein's modified method [9] was used in the work described in this paper.

Now according to Kratky and Sekora [12] and to Preston and Kapadia [10] stretching of viscose does not induce crystallisation to any extent. From these results it would seem that the number of accessible hydroxyl groups is reduced by orienting the molecules in the direction of the fibre axis, but that no crystallisation of amorphous cellulose accompanies the change.

The crystal lattice of cellulose can undergo certain changes. Native cellulose (cellulose I) can be converted into cellulose hydrate (cellulose II), and this form can undergo further change into cellulose IV, a type very similar to native cellulose. It is not clear yet to what extent these various forms of crystal lattice affect the sorptive capacity of cellulose. In this connection it should be mentioned that Howsmon [6] has introduced certain refinements which make allowance for the effect of size and type of crystallite structure when calculating the percentage accessibility of hydroxyl groups from moisture regain data.

On repeating work reported by Meyer and Badenhuizen [13] on the conversion of cellulose II to cellulose IV by heating in glycerol at 250°c., it has been found that a considerable decrease in sorptive capacity of cotton accompanies the change, but the X-ray photograph shows a smaller increase in definition than was expected.

TABLE II

	Sorptive Capacity (Ba activity No.)
Mercerised thread	160
Mercerised thread heated in glycerol at 250°c.	98

P. H. Hermans [14] has reported that, in the conversion of regenerated cellulose to cellulose IV by the same treatment, the decrease in sorptive capacity which accompanies the change is greater than would be expected from the comparatively slight increase in density. It seems probable, therefore, that the decrease in sorptive capacity might be caused by a combination of crystallisation of amorphous cellulose, conversion of the crystal lattice from cellulose II to cellulose IV, and of a change in the amorphous regions not detected by X-rays.

This particular problem of determining whether or not the type of crystal lattice influences the sorptive capacity is complicated by the fact that change from one type of lattice to another may be accompanied by changes in crystallite size and in percentage crystallinity. Furthermore it is possible that changes in the sorptive capacity of amorphous cellulose may accompany crystal lattice transformations, and yet these changes be of such a nature as not to appear as a change in crystallinity on any X-ray photograph.

While it has been shown that the small water molecules can penetrate into the cellulose II lattice [15], the larger dye molecules cannot enter, and it is therefore unlikely that the type of crystal lattice will have any effect on the equilibrium value at dyeing.

On this basis the treatments to which cotton is subjected prior to dyeing have been examined to determine whether or not they bring about any of the changes that are known to affect the sorptive capacity.

MERCERISATION

Treatment of cotton fibres with strong sodium hydroxide solutions brings about several changes. The proportion of amorphous cellulose is increased, if the cotton is allowed to shrink freely the degree of

orientation is decreased, and thirdly the crystal lattice is altered. The net effect of these alterations in fine structure is the well known increase in sorptive capacity.

The increase in amorphous cellulose can be detected in X-ray photographs, and the results reported in the literature [7,16] indicate that the amorphous content is increased from 30% to 50% on mercerisation.

Stretching orients the cellulose molecules, which is shown on X-ray photographs as a shortening of the arcs on the equatorial axis, and this results in a diminution in the sorptive capacity (cf. Table I). X-Ray photographs also show the effect of mercerisation upon the crystal lattice. The change is quite distinct.

TABLE III

Temperature of Wash after Mercerising (°F.)	Sorptive Capacity (Ba activity No.)
70	191·0
142	175·5
160	170·8
180	166·0
200	159·8
212	155·1

In a commercial mercerising process, the strong sodium hydroxide is washed out of cotton by hot water, and it has been found that the temperature of wash affects the sorptive capacity of the product [17]. The results obtained in a series of tests carried out during this work are shown in Table III.

Hess and Gundermann [18] found that a partial reversion to cellulose I was obtained by washing sodium cellulose I with water at 100°C., and recently it has been shown that mercerisation of viscose followed by washing in boiling water results in a crystallisation of amorphous cellulose [19]. It seems likely that the decrease in sorptive capacity is caused by such crystallisation.

From these experiments, it is obvious that attention must be paid to regularity of temperature of wash after mercerising, otherwise variation in the sorptive capacity may result.

BLEACHING

As far as can be judged from experiments that have been made, there are two stages in the bleaching process which bring about changes in the sorptive capacity of mercerised and unmercerised cotton. These stages are kier boiling and drying after bleaching. Hypochlorite and acid treatments do not seem to cause any serious changes in the fine structure provided they are applied correctly; but should they be applied wrongly, chemical damage occurs, and oxycellulose and hydrocellulose are formed.

KIER BOILING

Kier boiling causes a reduction in the sorptive capacity of cellulose of approx. 7% in the case of unmercerised cotton and of approx. 8–10% in the case of mercerised cotton.

When studying the effect of kier boiling upon the sorptive capacity of cotton, one is faced with the difficulty of interference from the natural impurities which are present in cotton. In the case of mercerised articles, a way round the difficulty is to purify the cotton prior to mercerising and then carry out experiments on the purified mercerised material. An alternative method is to use some means of removing the impurities after mercerising which will cause little or no change in the fine structure [9]. Both methods have been used and the agreement between them is good (Table IV). With unmercerised cotton, only the latter method, the less certain of the two, can be used.

In addition to measuring the sorptive capacity of the samples, X-ray photographs of a number of them were taken. Analyses were also made of the strength of caustic soda in samples of kier liquor drawn at different times during boiling.

TABLE IV

	Sorptive Capacity (Ba activity No.)
(*a*) Raw cotton	133
(*b*) Grey cotton, impurities removed by mild treatment (1% olive oil soap + 0·2% Na_2CO_3, boil for 1 hr.)	100
(*c*) Grey cotton kier-boiled, then given mild treatment (*b*)	93
(*d*) Grey cotton mercerised, and impurities then removed by mild treatment (*b*)	148
(*e*) Grey cotton mercerised, kier-boiled, and then given mild purifying treatment (*b*) ...	134
(*f*) Cotton purified, mercerised, and then given mild purifying treatment (*b*)	150
(*g*) Cotton purified, mercerised, kier-boiled, and then given mild purifying treatment (*b*) ...	136

The effect of kier boiling on the sorptive capacity of cotton is of practical and theoretical interest. From the practical point of view, it is essential that the extent of the change in sorptive capacity is regular throughout a bleaching lot, and if possible between one bleaching lot and another, otherwise unlevelness on dyeing may result.

From the theoretical point of view, the cause of the maximum in the caustic soda–time curve during kier boiling of mercerised cotton (Fig. 1) is of interest. The increase in concentration of caustic soda can be explained as follows. When cotton is immersed in the kier liquor, it absorbs caustic soda. During the boil, the sorptive capacity of the cotton is reduced, so that, if the change takes place quickly, an increase in the concentration of caustic soda in the kier liquor is observed. The results in Table IV indicate that a decrease in sorptive capacity occurs during the boil.

In an attempt to determine the nature of this change, samples of cotton were X-rayed before and after kier boiling. The X-ray photographs showed that there was an increase in crystallinity when both unmercerised and mercerised cottons were kier-boiled.

There are therefore three possibilities—

(1) That, since mercerised cotton is more reactive than unmercerised cotton, it crystallises more rapidly, with the result that a change in

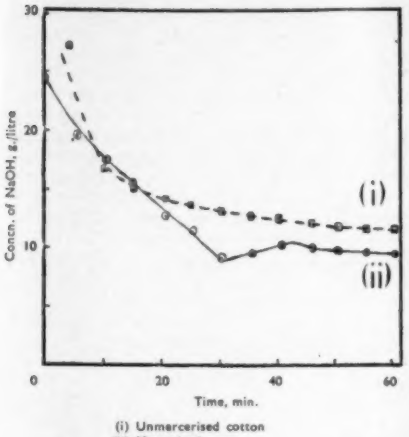

FIG. 1—Change in Strength of Liquor during Kier Boiling

sorptive capacity takes place quickly enough to effect a change in the concentration of caustic soda in the kier liquor.

(2) That a change, not detected by X-ray examination, takes place in the amorphous structure of mercerised cotton, producing a rapid drop in the sorptive capacity.

(3) That the effect is produced by a combination of (1) and (2).

HYPOCHLORITE AND ACID TREATMENTS

No indications have been obtained of controlled hypochlorite treatment having any measurable effect upon the sorptive capacity. The same result was also found in the case of acid treatment, although it should be noted that strong acid treatment does lead to substantial reduction in the sorptive capacity. This has been reported by a number of workers (Howsmon[6], Ingersoll[20], Hermans and Weidinger[20]). The results obtained in this investigation are in agreement with those reported in the literature and are shown in Table V.

The reduction in sorptive capacity of cellulose as a result of hydrolysis by strong acid is attributed to a crystallisation of part of the amorphous region together with a removal into solution of amorphous cellulose of low molecular weight.

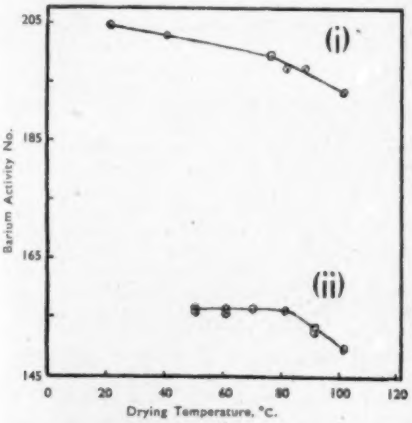

FIG. 2—Effect of Drying Temperature on Sorptive Capacity

TABLE V

		Sorptive Capacity (Ba activity No.)
(*a*)	Grey cotton, impurities removed by mild treatment (Table IV) ...	100
(*b*)	Grey cotton treated with 5N-HCl at 50°C. for 90 min., then given mild treatment	83
(*c*)	Grey cotton mercerised, impurities removed by mild treatment ...	148
(*d*)	Grey cotton mercerised, treated with 5N-HCl, then given mild treatment	120
(*e*)	Cotton purified, mercerised, and then given mild purifying treatment	150
(*f*)	Cotton purified, mercerised, treated with 5N-HCl, then given mild purifying treatment	118

DRYING OF COTTON AFTER BLEACHING

It has been known for a long time that drying reduces the sorptive capacity of both mercerised and unmercerised cottons. Tests have shown that, in the case of cotton mercerised under tension, there is little difference in the sorptive capacity of samples dried at room temperature and at 80°C. In the case of cotton mercerised without tension, the decrease in sorptive capacity with increasing drying temperature is greater per degree rise in temperature above 80°C. than per degree rise in temperature below 80°C., as shown in Fig. 2.

It has been reported that crystallisation of amorphous cellulose takes place on drying[21]. This would account for the reduction in sorptive capacity that results from drying, although this reduction could be caused by a partial collapse of a distended amorphous cellulose structure without any increase in crystallinity. It has also been suggested[22] that the water–secondary alcoholic group complex in cellulose has only a transient existence at temperatures above 80°C. If this hypothesis is correct, water combined with secondary alcoholic groups, viz. two-thirds of the total chemically combined water, would be removed above 80°C., and a partial collapse of the amorphous cellulose structure would take place. This could account for the larger decrease in sorptive capacity when samples were dried above this temperature.

Irregular drying, particularly above 80°C., leads to differences in sorptive capacity, and such differences give rise to unlevel dyeing.

RATE OF DYEING

As well as considering the effect of sorption equilibrium values of cotton on levelness of dyeing, account must also be taken of the effect of variation

in sorptive capacity on rate of dyeing [10]. Should the time of dyeing be long enough for equilibrium to be reached, then a close parallel will be obtained between the amount of dye absorbed and moisture regain, barium activity number, etc. If the dyeing time is short, equilibrium will not be reached, and as a rule differences in sorptive capacity will be exaggerated.

CONCLUSIONS

To sum up, it has been shown that cellulose is fairly reactive and readily undergoes change, and that any irregular treatment which the cotton may receive during mercerising, kier boiling, and drying can cause variations in the sorptive capacity of the material as a result of changes in the fine structure, such as percentage crystallinity, orientation, type of crystal lattice, and size of crystallite. These variations in sorptive capacity can give rise to unlevel dyeing.

TOPOGRAPHICAL EFFECTS

It should be pointed out that, in the above cases, only the overall average condition of the fibre has been considered, and, while the treatment given to the problem might satisfactorily account for certain properties of the fibre towards dyes, consideration must be given to topographical effects.

It has been shown by Preston [23], Elöd [24], and others that viscose consists of a skin and a core which have different reactivities towards dyes. The structure of cotton is complex, there being a central canal surrounded by many layers of secondary cellulose, which in turn are surrounded by layers of primary cellulose, and finally there is an outer sheath of wax, gums, protein, and pectic substances.

Experiments carried out recently in America have shown that the degree of polymerisation of primary and secondary cellulose in cotton differs, being 5000–6000 and 10,000–11,000 respectively. It has also been suggested that the orientation and crystallinity are different in the primary layer [25].

The effect produced by mechanical abrasion provides an example of the differences in "activity" of the various layers of cotton. It is quite well known that abrasion of cotton surfaces results in exposure of a more active layer of secondary cellulose [26]. Advantage is taken of this in the Congo Red staining test for distinguishing mechanical damage from chemical damage.

* * *

The author wishes to thank Dr. N. F. H. Bright and Dr. S. Fordham of the Research Department of Imperial Chemical Industries Ltd. (Nobel Division) for taking X-ray photographs of samples and for helpful discussion of the results. Thanks are also due to the directors of J. & P. Coats Ltd. for permission to publish this paper.

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Recent Developments in Fluorescent Lamps, with particular reference to Colour Problems

S. T. HENDERSON

Joint Meeting of the Midlands Section with the British Association of Chemists held at the School of Arts and Crafts, Derby, on 21st March 1951, Mr. A. J. Wesson in the chair

A brief survey of the factors influencing fluorescent lamp utility is followed by an account of the phosphors used, with particular reference to their emission bands and the specification of the resulting colour properties of lamps by spectrophotometry and colorimetry. The problem of adequate colour rendering is discussed with examples, in particular the attempts made to improve rendering at the red end of the spectrum.

INTRODUCTION

In this paper we propose to begin with a very brief account of the fluorescent lamp, mentioning the features in which improvements are possible or are being made; and in particular the subject of phosphors, since this is of chemical interest and is besides of primary importance. Next we shall consider spectral emission curves and colour specifications, and in fact take a general view of the varied and often conflicting ideas which make up the subject of "colour". Finally, we come to the urgent but difficult matter of colour rendering, and show what is required and what is being attempted, emphasising here, perhaps, the difficulties rather than the achievements.

THE FLUORESCENT LAMP

The low-pressure fluorescent lamp is essentially an arc in mercury vapour between electrodes, which may be of the "cold" type using high-voltage supply, or "hot" as in the mains-voltage lamp. A rare gas filling, usually argon, permits easy starting, but in normal running the mercury arc alone is seen, and the resonance radiation at 2537 A. in this arc is the chief exciting agent for the fluorescent powder on the lamp walls. The desirable properties of the lamp are efficiency, good maintenance of light output, long life, and suitable colour, not necessarily in that order. Colour depends almost entirely on the phosphor. Long life is largely a matter of cathode life and carefully controlled lamp processing, but is clearly useless without a phosphor of good maintenance. The importance of the phosphor in efficiency is evident; and thus for several reasons the major effort in improving fluorescent lamps has lately been concentrated on the phosphor. There seems to be little hope of revolutionary changes in cathode design, in composition of gas filling, or in processing techniques, though much has been done in these directions. An improvement in the conversion of electrical energy to 2537 A. radiation is greatly to be desired, but no one seems to have any ideas on that subject. We may, therefore, usefully consider the phosphor in more detail, since it affects nearly every aspect of lamp production and performance.

THE PHOSPHOR

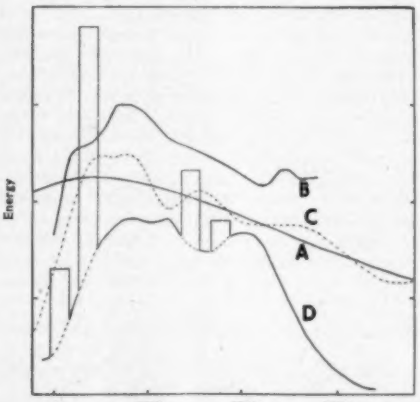
The phosphors used in fluorescent lamps are inorganic solids, very often including a Group II metal as their main metallic ion, with anions which vary very widely. Thus there are silicates, tungstates, phosphates, borates, arsenates, germanates, titanates, etc., and like the naturally occurring minerals these synthetic phosphors are often non-stoichiometric in composition. In passing it may be remarked that, while natural minerals provide examples of fluorescent solids, and have suggested classes of compounds for laboratory investigation, artificial preparation usually gives materials of greater light output. In general these "matrices" are colourless, and are made by thermal synthesis from oxides, etc., sometimes with added fluxes, in such a way as to make them recognisably crystalline by X-ray diffraction. Glasses are sometimes luminescent, but are not of any particular importance in lamps. Visibly crystalline powders, which could often be formed by longer heat treatments, are undesirable for the purpose, because coating a thin layer inside the lamp would be made more difficult by large grain size. Phosphors have in most cases an essential ingredient known as the activator, which is usually a foreign heavy metal present to the extent of, say, 0·01–5%, and without which the characteristic fluorescence is not developed. These activators have to be incorporated by heat treatment, generally during the formation of the matrix itself; they include a great variety of metals, but manganese is pre-eminent for the phosphors we are considering.

From the chemist's point of view the main interest of these materials lies in their extraordinary sensitivity to impurities, particularly metals such as copper, iron, cobalt, and nickel, though these may, contrariwise, function as activators in certain cases. In general the fluorescence chemist has to work to a purity of about 10 p.p.m., and this involves considerable effort when powders are made on the large scale. Since fluorescence of the completed phosphor is invariably a more sensitive test of contamination than any intermediate control during its preparation, this manufacture is also apt to cause much anxiety. An inefficient or contaminated phosphor is rarely worth attempts at regeneration, and though the basic raw materials are often fairly cheap, a large amount of labour goes into the finished product. The world output of powders for fluorescent lamps is now of the order of 500 tons per annum, costing roughly £1,000,000, so that the industry is an important one[1]. There is no space to do more than mention the sulphide phosphors, which are used on a much smaller scale for cathode ray tube screens; these phosphors require a much higher degree of purity even than the lamp phosphors.

LAMP COLOUR

The property of these phosphors with which we are mainly concerned is their spectral emission. Speaking generally, all of them exhibit wide, structureless bands, which can be represented by Gaussian error curves when plotted on a frequency instead of a wavelength scale. There may be one or more bands derived from one matrix, or an apparently simple band may be resolvable into separate sub-bands of Gaussian form[2]. The bands may lie in almost any part of the visible spectrum, occasionally in the ultra-violet or infra-red. These facts have much interest in connection with the mechanism of fluorescence, namely the electronic transitions which produce the light emission, but for our present purpose we have to consider the less easily measured effects which the light produces through the eye and brain.

The lamp technologist produces his different colours by mixing phosphors, and with the wide variety at hand an immense number of combined emission curves can be obtained. This, of course, is additive mixture, not subtractive as used by most colourists. One other significant fact has to be remembered: it is the invariable presence of the mercury spectrum in addition to the phosphor bands, giving the kind of energy distribution



A ... 6500°K. Black body C ... Source C
B ... 6500°K. Sky D ... Northlight fluorescent lamp

FIG. 1

shown in the spectrophotometer curves in Fig. 1 and 4. The mercury lines are conventionally shown as bands 200 A. wide; in this way their energy can be represented by the areas enclosed by these blocks, on the same scale as the energy of the phosphor emission (ordinates represent energy per unit wavelength). The energy distribution is entirely different from those of other light sources, natural or artificial, and it is tempting to suggest that it accounts for most of the unusual properties of fluorescent lighting, including some of the colour rendering difficulties. However, it does not prove very easy to fix the responsibility in this way.

Before discussing the subject of colour measurement, the use of external or internal filter coatings for obtaining special effects in fluorescent lamps may be mentioned. These filters seriously lower the efficiency for which fluorescent lighting is notable, but in theatre work it is necessary to have more saturated colours, which if desired can also be mixed to give innumerable less saturated hues. In normal lamps the mercury spectrum has a large desaturating effect, which these filters largely correct. There is still an advantage in efficiency in the filtered types compared with incandescent lamps using coloured "gelatins".

We have seen that the complete emission curve of a light source can be drawn by spectrophotometry. This appears to give complete and objective data on the lamp. But when the effect on the eye is examined, we find that colour perception is not a purely physical matter, and it is difficult to say from the spectrophotometric curve what colour the eye will "see". Also, an infinite variety of lights with different curves can give the same sensation. As an example, consider a sodium flame, and an orange–yellow paper in sunlight. These could look exactly alike, although their curves could hardly be more different; for the flame has the D lines only, and the paper reflects a wide band covering green, yellow, and red— but crossed by the Fraunhofer lines, i.e. lacking the D lines.

Arising from this psycho-physical aspect of colour, we have a second type of colour measurement, which is to find the chromaticity (or colour appearance) in the international units X, Y, Z, a well known technique with the advantage of two-dimensional plotting of the results and reasonable independence of observer variations. The procedure mostly involves the use of trichromatic colorimeters for visual matching, at any rate in this country, though photoelectric methods are also used. In the U.S.A. they prefer to calculate the chromaticity from the spectrophotometric curve, which is not the purely physical operation it might appear to be, since the calculation uses tables of data derived from the responses of the "average eye". In both cases obtaining the chromaticity is irreversible. Any point on the X, Y, Z diagram represents an infinite number of different spectral distributions, all looking alike to the eye. Dyers will appreciate that this is an example of the phenomenon of metamerism, which is fundamental to colour vision and therefore affects visual colorimetry, to the detriment of that rather exhausting pursuit. The latest type of visual colorimeter, Donaldson's instrument with six primaries[3], ensures a better agreement in spectral distribution between the two halves of the field, but even this cannot be very close in the presence of mercury lines.

The only other basic principle to be stated is that non-self-luminous objects are "coloured" only by virtue of their illumination by a light source, and the light they reflect to the eye is physically described by the product of their spectral reflectance curve with the emission curve of the source at each wavelength. As a result, we cannot tell what colour an illuminated surface will appear to be,

even if we know its spectral reflectivity, if we also know *only* the chromaticity or colour appearance of the light source.

COLOUR RENDERING

This last sentence expresses the essence of the colour rendering problem. In its practical aspect, we find that the earlier attitude of satisfaction with fluorescent lighting because it was novel, efficient, and able to give a light of any colour is now being replaced by criticism of the appearance of the objects illuminated. There are still many industrial applications in which efficiency is more important than colour rendering, but the change which is proceeding in the more social and domestic fields may well be demanded in industry before long.

On the subject of using physical data for colour rendering specifications it is necessary to mention the "eight-band diagram", which in effect converts the spectral energy curve of a source into terms of visibility by use of the "equal energy luminosity curve for the average eye", and then integrates the luminance in each of eight sections comprising the visible spectrum. Values are tabulated or represented graphically as a histogram. This process successfully conceals the mercury lines and does give a rough general idea of the emission, but it has many drawbacks in spite of its elaborate theoretical and practical basis[4], especially in its suppression of the ends of the spectrum. In its present form it has not gained general acceptance, and further studies are in progress.

It is important to note that the sensitivity of the eye is at a maximum in the yellow–green region, falling to very low values in the blue and red. If the energy distribution curves are corrected to visibility (or luminance), the importance of the ends of the spectrum seems to be reduced to negligible dimensions. In practice these parts of the spectrum are important in colour rendering, as will appear below, particularly in regard to the red end. Fig. 2 gives the curves for a lamp containing calcium silicate phosphor, with ordinates representing energy and luminance respectively. The lamp is a slightly bluish pink in colour, which might be guessed from the energy curve, but the interpretation of the luminance curve is not so easy, though this shows the eye responses in their correct relationship. The virtual disappearance of the powerful blue and violet lines is particularly misleading in the luminance curve. For clearer presentation the other curves are given with energy ordinates.

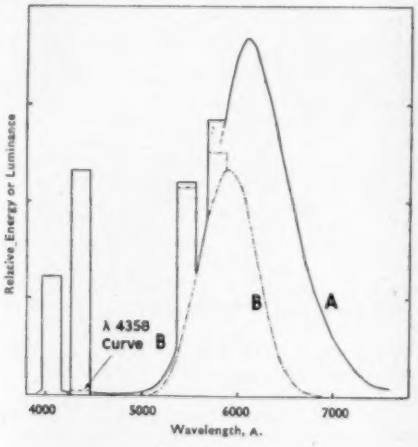


FIG. 2— Fluorescent Lamp (Rose)

We may now consider some practical examples of colour rendering problems, beginning with a fairly simple one in which there is an established standard of comparison. This is the accurate colour matching carried on by colour printers, dyers, paper manufacturers, and others. North skylight, although by no means a constant source in intensity or spectral composition, is used for the purpose, nominally at an equivalent colour temperature of 6500°K. Fig. 1 shows (Curve B) one of many curves published for this kind of light, and also (Curve A) the distribution for a complete radiator or "black body" at 6500°K. calculated from the Planck equation. Curves C and D give the measured distributions for the very similar source C used in colorimetry (comprising a tungsten lamp at 2848°K. and liquid filters), and for the equivalent fluorescent lamp. The wide differences in distribution shown in Fig. 1 suggest that differences in colour rendering will occur, and in fact these are found in critical work, but the Northlight lamp does provide a fairly satisfactory substitute for skylight in most cases. A better source is a combination of under-run tungsten lamps with fluorescent tubes coated with magnesium tungstate phosphor. The balance between these has to be maintained very carefully, which may be difficult in practice; metameric green cloths may be used as an aid to setting up the combination to the required colour.

Serious trouble for the lamp designer really begins, however, when the lamp colour moves away from near normal daylight towards warmer colours, particularly if the chromaticity falls below the "black body line". This is the locus on the chromaticity diagram of the colour appearance of incandescent black bodies at various temperatures. Colours a little below this line are more red or purple in hue than the black bodies, and they have been found acceptable for locations where efficiency is less important than appearance— for home use and for shops and restaurants. Incandescent lighting has long been used in these surroundings, and the distortions it introduces are mostly accepted without comment, although they are often quite serious. The reason for this acceptance is one of the mysteries of the subject. It may be due to some visual mechanism, and its reaction to the smooth spectral distribution characteristic of natural daylight and artificial illuminants from firelight to incandescent tungsten. No doubt eye adaptation helps by reducing the gap between daylight and incandescent light, but this subject cannot

be discussed here at any length. There also seems to be some special significance in the location of a colour on the black body locus, unless this is merely a matter of inherited visual experience. In the case of the American "warmtone" or "warm white" lamp, with a chromaticity the same as that of incandescent tungsten, it is found that this is accepted more readily for domestic and similar purposes than other fluorescent types. This may be because it blends well with tungsten lighting; it cannot be due to "black body" distribution of energy.

The distortions arising from fluorescent lighting are different from those caused by incandescent lighting, and as a result a good deal of criticism has been made, not always justifiably, and not often precisely enough to help the investigator. Perhaps the multiplicity of colours available has added somewhat to the confusion. What is required may be stated generally thus: a light source must not only be a pleasant colour to look at, but it must not make objects peculiar in appearance, particularly if they are complexions or foodstuffs. This is not at all exact, and yet it is a difficult requirement to fulfil, for everyday objects exhibit an infinite variety of reflectance curves, and no single lamp is likely to be satisfactory for everything. For instance, requests have been received for advice on the lighting of such things as diamonds and champagne, and in less expensive circles, cups of tea and slices of beetroot. The degree of accuracy or "naturalness" required for these objects is of course quite indefinite and may vary from one observer to another; we cannot imagine standards of acceptable approximation in colour rendering without great complication. A "duplication index" for this purpose has indeed been derived by the U.S. Bureau of Standards, using chromaticity determinations on three pairs of metameric cloths illuminated alternately by the lights under comparison; but the procedure is elaborate and arbitrary. For the present, therefore, methods of trial and error are largely used in attempts to produce new lamps and in appraisal of colour rendering. Compared with the immediate judgments of the eye, our lengthy processes of physical measurement seem unduly laborious, especially as they do not yet lead to convincing reasons for the instinctive favourable or adverse judgments.

A sort of consumer research has been carried out on the matter, in particular in the U.S.A., in which observers are presented with alternative lightings of all kinds of objects, seen singly or in groups. Voting by large numbers of observers for their preferences gives a mass of data, which are analysed and interpreted as "significant trends"[5].

The "colour box" demonstrated at the lecture (see appendix) shows the type of experiment, which can be done very conveniently in this equipment. So also can the accurate comparison of nearly identical light sources. But there are objections to the method when preferences are being studied.

A rapid choice made in this way will not necessarily be a reliable one; it may not be easy to "live with". A more weighty criticism is that judgments about the appearance of objects are usually not made with a control, but memory provides the standard of comparison—a very uncertain standard. The difficulties are accentuated by the fact, briefly mentioned above, that previous exposure of the eye to light, whether coloured or "white", may seriously affect its response to the next coloured object seen. That is, eye adaptation will alter colour perception, and this must be the usual condition in everyday life[6].

RED DEFICIENCY IN LAMPS

From the above rather discouraging outlook we may turn to one well defined problem, arising from a fairly obvious defect in the fluorescent lamp, which is now approaching its solution. This defect is that nearly all the phosphors emitting towards the long-wavelength end of the spectrum have their peak in the yellow or orange and fall off sharply in the red. This is in marked contrast to skylight, sunlight, and incandescent light, and consequently deficiency in red (or inadequate red rendering) has been recognised for a long time in fluorescent lamps. Much effort has been spent on the search for new phosphors of longer wavelength emission. Unfortunately, few are available and they have various drawbacks. The best so far in use in this country is probably calcium metasilicate activated by lead and manganese, an example of sensitised fluorescence in which lead produces an optical absorption band for the 2537 A. radiation[1,7]. The fluorescence so excited has a peak near that of the zinc beryllium silicates which were formerly used, but were abandoned a few years ago because of their poisonous nature. Cadmium borate activated by manganese has a yellow–red emission band with a peak near 6200 A. compared with calcium silicate at 6100 A., but the borate tends to be unstable in use.

In the U.S.A. lamp makers have recently improved on the colour rendering of their halophosphate powders by adding or substituting other phosphors of rather lower efficiency but better red emission, and calling the result "de luxe". It is pleasant to record that we in this country were previously producing lamps of similar colour rendering characteristics, though they were not called "de luxe"! Incidentally, the halophosphates were discovered here[8]; they are a class with a wide range of fluorescent colours given by changing ratios of intensity in different emission bands from the same matrix. They are widely accepted for their efficiency, but are deficient in red emission by comparison with the phosphors mentioned in the last paragraph. Fig. 3 gives the curves for two lamps of the same chromaticity (omitting mercury lines), showing the worse red rendering to be expected from the halophosphate.

It is believed that the American "de luxe" types use calcium silicate[1,7], or calcium orthophosphate activated by cerium and manganese[9]. This latter phosphor requires reducing conditions in preparation and processing to keep the cerium tervalent, when it acts similarly to lead in calcium silicate phosphor, but this requirement is unsatisfactory in manufacture. Other possibilities are magnesium germanate activated by manganese, a very expensive material with a magnificent fluorescent

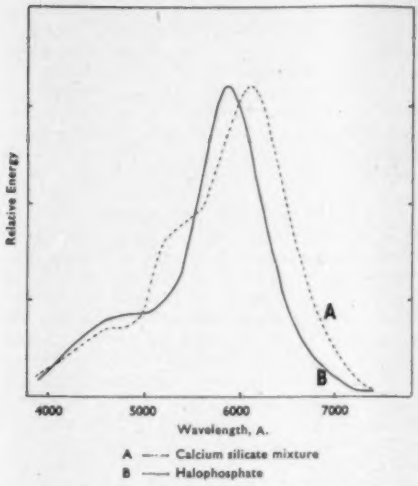


FIG. 3— White (3500°K.) Fluorescent Lamp (mercury lines omitted)

colour[10]; and the similar arsenate[11], which is plainly not a material to be made lightheartedly in hundredweight lots. Fig. 4 gives the emission curves of some of these without mercury lines, and for clarity arbitrary levels are chosen for the peaks. In terms of absolute energy or of visibility these red materials are all much less efficient than calcium silicate, hence the significance of band position at this end of the spectrum. Fig. 4 includes a very unusual material in alumina activated by a trace of iron and containing lithium[12]. The fluted structure on the long-wavelength side is difficult to measure, but seems to be genuine. It is reminiscent of chromium-activated alumina under cathode-ray excitation.

By using the germanate or arsenate phosphors it is possible to obtain a light of the same appearance as incandescent tungsten, but which makes red objects even more prominent than under the latter. This raises the question of whether it is possible, or desirable, to give the impression of good overall colour rendering by overemphasis of the more prominent colours, say red and green, with comparative neglect of yellow and blue. In default

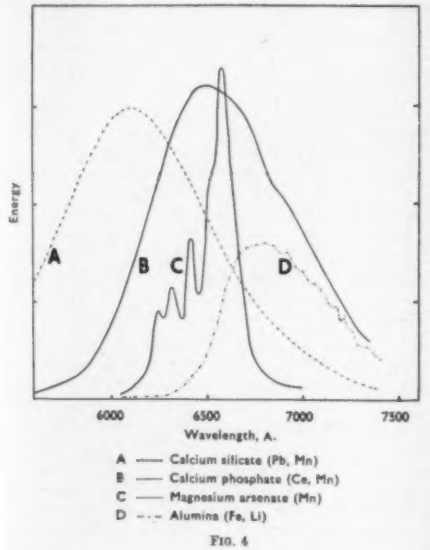


FIG. 4

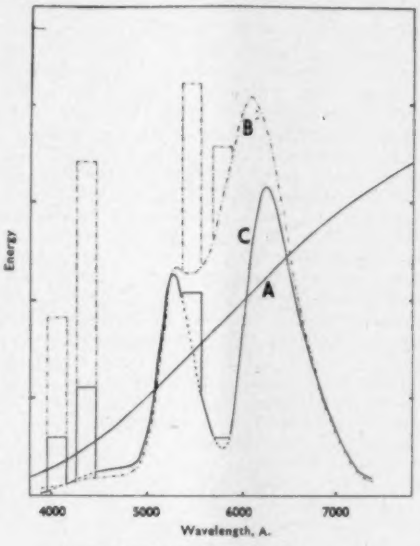


FIG. 5

of a satisfactory theoretical approach, a great deal of practical experience will be required before these matters can be settled. An example of the value of suppressing the yellow is given below.

Meanwhile the search for new phosphors goes on, usually by tests on great numbers of preparations in some likely field, with variation of the many factors in composition and treatment which are known to affect the results. Occasionally, the patient chemist is rewarded by a new phosphor, and there seems no reason to doubt that many more will be discovered in the future.

Another interesting line we have begun to explore on the more physical side is the importance of marked unevenness in the spectral distribution. Tungsten light brings out the reds better than a matching combination of blue (magnesium tungstate), green (zinc silicate), and pink (calcium silicate) tubes, which is not surprising, since the red emission of the pink lamp ends near 7000 A. But if the pink tubes are provided with red colour filters

and the combination again matched to tungsten, the reds are as prominent, if not more so, under the fluorescent mixture by comparison with the incandescent source. Fig. 5 shows the spectrophotometer curves for the combinations and the black body distribution of the tungsten; matches were made at equal brightness, but for clarity in the figure one curve has been expanded vertically. The main difference to be noted is the strongly peaked distribution of the combination with red filters. Another example of the same kind has been mentioned in connection with the germanate and arsenate phosphors, which in spite of low efficiency produce an impressive enhancement of the reds. The isolation of the red band (or the absence of orange–yellow) thus appears to be a major factor in the improvement of red rendering, but the subject may also be worth further study from the angle of the spectral distribution of retinal receptors[13].

OTHER TOPICS

Little has been said in this paper, and little work has been done, on the importance of the mercury lines in the fluorescent lamp spectrum[14]. Normally the violet and blue lines are more prominent on an energy basis (but not in luminance, see Fig. 2) than the green and yellow, which often lie on prominent phosphor bands. Partial removal of the violet end of the spectrum (and any ultra-violet) by pale yellow filters might be helpful in special cases; thus the fluorescence of fats imparts a greenish hue, and an improvement in appearance might be made by discreet filtering. In practice this is not very easy. Another problem is the proportion of mercury light in the total emission, which varies with temperature and hence with loading of the lamp. This causes chromaticity differences if the same phosphor is used in lamps of different sizes.

There are numerous other related subjects which have not been covered. There would, for example, be interest in the development and trial of phosphors emitting in the far red, or phosphors with narrow bands to reinforce special regions; then several good ultraviolet-emitting powders are known, but their performance would be much improved if there were some way of eliminating the unwanted mercury lines which add visible light to the ultra-violet emission. The problem of selective removal of mercury lines seems insoluble, but perhaps there is no harm in stating it. This paper has, in fact, done little more than try to state the problems of colour as they appear to the lamp maker, and to show that, though their appreciation is a matter of physics, their solution lies very largely in the hands of the chemist.

* * *

The author is indebted to his colleagues for help in the work, and for criticism of the paper; in particular to Miss M. B. Halstead for spectrophotometric and colorimetric measurements. Thanks are also due to the directors of Thorn Electrical Industries Ltd. for permission to publish.

Appendix

COLOUR BOX

The "colour box" consists of a sheet aluminium box with a thin vertical partition dividing it into two compartments, each 18 in. wide by 30 in. deep by about 36 in. high. Each half can be independently lit by any desired combination of lamps at suitable brightness levels, and the results compared by an observer on the line of the partition, which is movable so that flat patterns can be exposed with part in each compartment.

Daylight can be admitted by opening one or both sections of the roof towards a suitable window behind the box. In the roof of each half is a detachable tray of six fluorescent lamps, 2 ft. 40 watt in size. Of these one pair is in series in one circuit, a second pair is in series in a second circuit, and the remaining two are in separate circuits. Each circuit can be dimmed in nine steps from full brightness to a low value by means of series resistors, the lamp cathodes being kept at emitting temperature throughout by separate supplies. There are thus four independent dimming circuits for each half of the box. Four bayonet sockets are located near the corners of each tray of fluorescent lamps, and 75-w. pearl incandescent bulbs are used here when required. In one half the incandescent lamps may also be dimmed to any level. All control gear is located outside the colour box in two units, one with chokes and transformers, the other with switches and dimming controls on a panel. The interior walls and floor have been painted experimentally with a battleship grey paint. This gives very great sensitivity in adjusting colours to the same appearance, but its colour neutrality is in doubt. Observations on the relative colour rendering properties of two light sources, one in each box, are made at equal luminance as far as possible.

PATTERNS

The patterns are sheets of coloured paper about 50 cm. square. Each has one half covered with parallel strips of green (50 cm. × 0·8 cm.) placed with 1·2 cm. spaces between the strips. The other half continues with the same pattern of strips but of a royal blue colour. The first sheet is of a canary yellow colour, the other of a bright orange. These colours may be referred to by their equivalents in *B.S.* 381C:1948. The backgrounds are close to No. 309 and No. 557, the blue strips on each approximately between No. 166 and 108, while the green strips are somewhat bluer than No. 280.

When viewed at close range either background is clearly uniform in spite of the two colours of strips on it. At a distance, however, the yellow between the blue strips changes in appearance to a pale, dull colour contrasting with the bright yellow seen between the green strips. The orange background shows an even greater contrast, becoming bright pink where overlaid by the blue strips.

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Discussion

Mr. A. J. WESSON: Why are twin tubes used in the Atlas Northlight matching lamp? Is it possible to run them singly?

Dr. HENDERSON: Twin tubes are used instead of single ones merely to produce more light from one fitting; there is no reason why a single tube should not be used (with the appropriate ballast).

The twin tubes are intended to be identical, not a specially matched pair.

Mr. A. S. WHELDON: My experience confirms that difficulty is sometimes found in matching reds by fluorescent lamps. Why should the problem be worse on rayon than on other fabrics such as wool?

Dr. HENDERSON: This may be due to some difference in reflectivity of the different fabrics, which may affect the saturation of the colour seen by the eye and so alter the discrimination possible. Incidentally, it is hoped that a Northlight lamp with better red rendering may be available later as a result of phosphor developments (other than those mentioned in the paper).

A MEMBER: Can any vapour be used to fill the tube other than mercury and argon? Why not neon to improve red emission?

Dr. HENDERSON: Krypton may be used with mercury, giving somewhat higher efficiency though at greater lamp cost. Neon has a spectrum which is an excellent source of red wavelengths, but the presence of mercury suppresses the neon spectrum completely. Neon can be used by itself, as in the uncoated cold-cathode tubing often seen. In hot-cathode lamps without mercury it suffers from the disadvantages of (1) high striking voltage, (2) lack of response of some phosphors to the resonance radiation at 736 A., and (3) very low efficiency of conversion of this radiation into visible light with any phosphor that is excited by it (about one-seventh as against one-half for mercury 2537 A.).

Mr. GIBSON: Why is the choice of phosphor restricted to inorganic materials when very many organic substances of high fluorescence are available?

Dr. HENDERSON: Organic substances are mostly fluorescent to longer-wavelength ultra-violet than the mercury resonance radiation, and besides, the processing of the lamp at roughly 400°c. would destroy these materials. Even if they survived into the finished lamp, the mercury vapour would no doubt react with most of them, and photo-chemical changes under ultra-violet radiation would also destroy them.

Mr. R. S. LOCKE: Particular reference has been made to 2537 A. radiation in the tube. Do such tubes give any other ultra-violet radiation? Also how does the ultra-violet output compare with that of the commercial tungsten lamp?

Dr. HENDERSON: There is a small output of longer ultra-violet, mostly in the 3650 A. region, and after partial absorption by the phosphor this is transmitted by the glass envelope (whereas 2537 A. radiation is completely absorbed). Compared with the ultra-violet emitted by a tungsten lamp of equal wattage, the amount is probably somewhat higher from the fluorescent lamp, but both are small compared with the ultra-violet in the equivalent amount of sunlight.

Mr. A. J. WESSON: Can an explanation be given of the illusion of colour change given by the striped material exhibited?

Dr. HENDERSON: These patterns (see Appendix) were developed after seeing similar ones demonstrated by Dr. W. D. Wright at his recent "Thomas Young oration" in London[15]. Against the suggestion that a fatigue effect is involved, it may be pointed out that the illusion is visible immediately; no appreciable time lag occurs. It seems to depend on overlapping of the images of adjacent stripes on the retina, and there is a critical distance for any observer beyond which the change appears. This may be altered by using lenses in front of the eye, or by changing the direction of the stripes if the eye is astigmatic. It would be interesting to achromatise the eye by a suitable lens system and see whether that removed the effect.

Mr. PARKINSON: Arising out of the reply to the last question, are we not making a fundamental mistake in assuming that the retinal rods are, in fact, stationary? Such a phenomenon would be more easily comprehended if we assumed a limited movement.

Dr. HENDERSON: I know of no evidence that the retinal receptors are other than fixed in position and colour sensitivity, though there is much difference of opinion on the nature of their individual colour response, grouping in the retina, and so on.

COMMUNICATION

Advances in the Application of Vat Dyes to Viscose Rayon Cakes

H. HAMPSON

The dyeing of viscose rayon cakes with vat dyes is discussed. The use of vat dye pigments of a particle size of approx. 2–3 μ. diameter for application by a pigmentation–reduction technique of dyeing is shown to produce satisfactory results. This low degree of aggregation of particles is achieved by regrinding commercial brands in a ball or pebble mill.

I— Introduction

The demand in recent years for improved quality of dyed yarn has focussed attention on the application of vat dyes to viscose rayon cakes. Apart from the saving in time and cost in reeling the cake yarn into skeins for dyeing, the advantages to be obtained by dyeing viscose rayon in cake form rather than in skein have been given by Whittaker [1] and might well be restated here— (1) One operator can supervise the winding of more dyed cakes than dyed skeins, whilst less time is taken to train a cake winder than to train a skein winder. (2) There is less winding waste. (3) Cake-dyed yarn is superior in quality to skein-dyed yarn. (4) There are fewer knots per cone. (5) The rate of production in weaving and winding is higher.

The dyeing of rayon packages raises all the problems of the cellulosic fibre–substantive dye system in an acute form. The dyer's first aim is to produce a uniformly dyed product. This is more readily achieved with well separated fibres or filaments, as in the case of jig dyeing of piece goods, than with a more densely packed mass of fibres, as in a viscose rayon cake. In the first case, no matter how rapidly the dyebath is exhausted, the chances of uniform deposition of dye on fibre surfaces are relatively high and the time required for perfect redistribution is short and largely independent of intrinsic dye-diffusion properties. In the dyeing of dense thick packages, exhaustion of the dyebath does not correspond with uniform distribution of dye; the dye is adsorbed relatively rapidly on and near the surfaces of the solid package, and redistribution depends upon the slow diffusion of dye into and out of filaments with high dye adsorption and into filaments unstained by dye.

II— The Dyeing Process

It has been shown by Boulton and Morton [2] that the dyeing of vat dyes in the alkaline leuco state under existing commercial conditions proceeds in two stages— (1) surface adsorption of the dye by part of the dyed material, which causes the dyebath to become rapidly and completely exhausted; (2) a process of levelling dependent for its speed upon the ease of diffusion of the leuco dye in the viscose rayon or other cellulosic fibre.

The first of these stages governs the "strike", i.e. the amount of dye adsorbed by the fibre in the first minutes of dyeing, and this amount is great with many vat dyes on viscose rayon, the dye liquor being commonly exhausted of dye in less than one minute. The second stage is very slow with most dyes, especially with the anthraquinonoid and benzanthrone types.

Boulton and Morton [2], Burgess [3], and Wilcock [4] give details of a test devised by Wilcock for determining the strike and subsequent levelling properties of a dye, the so-called strike–migration test. More recently details of a similar and other tests have been published by the Society's Vat Dyes Committee [5].

It is well known that both the strike and the rate of diffusion can be controlled to some extent by temperature. The lower the temperature of dyeing the lower the rate of exhaustion and, therefore, the more controllable the strike, whilst increase in temperature gives a higher rate of diffusion, leading to improved levelling properties of the dye after its deposition on the fibre.

1. RETARDING AGENTS

The use of retarding agents for controlling the rate of exhaustion of the dye has for many years been suggested as a means of producing level dyeings from unlevel-dyeing vat dyes, products of varying constitution having been recommended by dye manufacturers and others. It is suggested that these act by forming unstable addition compounds with the leuco vat dye which aggregate in the dye liquor, and as a result the rate of adsorption of the dye is lowered and a more level dyeing obtained following a more complete distribution of the dye throughout the mass of fibre [6,7].

Boulton [8] describes the action with sulphite cellulose waste liquors containing magnesium sulphate as follows—

> . . . in the alkaline condition of the dyebath [the magnesium sulphate] produces magnesium hydroxide, which is maintained in the colloidal state by the protective colloid, lignin sulphonic acid. Dye ions replace some of the hydroxyl ions in the lignin magnesium hydroxide complex and are held by this complex so that a dynamic equilibrium exists:
>
> Dye cellulose complex L.H. $\rightleftharpoons$ Dye in true solution $\rightleftharpoons$ Dye lignin sulphonate Mg complex R.H.
>
> Dye ions are freed progressively by the R.H. complex to take part in the dyeing. This theory has been amply proved by T. H. Morton who first suggested it.[9]

The restraining action of Dispersol VL (ICI) (a fatty alcohol–ethylene oxide condensate in water) with leuco vat dyes has been described by Speke [10]. These products are not true levelling agents, in that they will not transfer dye from a heavily dyed portion to a lightly dyed portion. They all act in a similar way in that they effect an improved levelness of the dyeing by reducing the affinity of the vat dye for the fibre, and so the exhaustion of the dye is controlled and dye is available for the least

accessible parts of the material. This restraining action is accompanied by loss of dye available for colouring the material, and further the degree of restraint varies greatly from dye to dye. Burgess [3] and Wilcock [4] have given figures for the loss in "colour value" of certain dyes when dyed in the presence of sulphite cellulose waste liquor.

Early attempts at the dyeing of viscose rayon in cake form on package-dyeing machines using the unmodified alkaline leuco vat with or without restraining or retarding agents were unsuccessful, the dye being exhausted on the inside or outside of the package according to the direction of flow, the middle of the cake being left white or only thinly dyed. It was possible to achieve a moderate degree of levelness by the use of large quantities of restraining agents with those dyes which are very sensitive to this restraining action. It is only possible, however, to obtain very thin shades by this means, which is inadequate for meeting commercial needs where all depths of dyeing in a wide variety of shades are wanted. Attempts were made to obtain heavy dyeings from highly restrained dyes by the gradual addition of a substance capable of counteracting the effect of the restraining agent. This technique was unsuccessful; immediately "neutralisation" point is reached, if there is any large proportion of the dye still unadsorbed, adsorption begins at as high a rate as normally.

2. ACID–LEUCO DYEING

Attempts were made to dye viscose rayon cakes by the use of the acid leuco compound of the dye (the vat–acid process [11]). These were also unsuccessful owing to the following factors—(1) instability and precipitation of the acid leuco compounds in general, especially in circulating dyeing machines; (2) low affinity for cellulosic fibres, requiring the addition of alkali to impart affinity, and the consequent incidence of normal leuco dye difficulties.

3. USE OF SOLVENTS

The use of solvents has been claimed as an aid for obtaining more level results with leuco vat dyes. One patent specification [12] describes the use of 20% (by weight) solutions of organic solvents, the solvents being lower alcohols containing not more than five carbon atoms in the molecule, preferably ethanol, and states that: "The effect of the addition of the said solvents is probably due to their increasing degree of dispersion of the alkali salts of the leuco base". A second specification [13] claims for the dyeing of wound packages of viscose or cuprammonium rayon the use of water-soluble aliphatic mono- or poly-hydric alcohols such as methyl alcohol, ethyl alcohol, propyl alcohol, glycols, and the like in concentrations less than 25% by volume, and advances the theory that the effect is due to restriction of the swelling of the fibres, which renders it possible to maintain the permeability of the wound package.

Reference to the use of solvents has been made by Whittaker and Wilcock [6] and by Boulton [8], the latter suggesting that the dissolved dye is in more nearly molecular solution, in which state it diffuses more rapidly.

The use of solvents for the levelling of vat dyes when dyeing viscose rayon cakes was investigated by Wilcock, Hampson, and co-workers, who found that many vat dyes were more easily levelled in the presence of solvents, such as methylated spirits, Carbitol (diethylene glycol monoethyl ether), and Butyl Carbitol (diethylene glycol monobutyl ether). Examples of the quantities required are a 25% solution of methylated spirits, a 10% solution of Carbitol, and a 3–5% solution of Butyl Carbitol, all by volume.

It was found that dyes varied in the time required to complete levelling and in the amount of solvent necessary. The rate of levelling increased with rise in temperature. Lists of dyes which level and which do not level when leuco-dyed from a 25% solution of industrial alcohol in water are given by Boulton [8]. Butyl Carbitol is preferred, since less bulk is needed and since it has a higher boiling point than methylated spirits, permitting the use of higher dyeing temperatures (80°c.) with dyes that are stable at that temperature, without loss of solvent due to evaporation.

Although the successful use of Butyl Carbitol–water dyebaths has been amply demonstrated, the system has not been attempted on a commercial scale for economic reasons. The solvents are costly in themselves, and it would be necessary to install solvent-recovery plants at a very high capital cost before dyeing with solvents could be undertaken at anything approaching existing vat dyeing prices.

4. USE OF VAT PIGMENTS

The next important step towards the successful application of vat dyes to viscose rayon cakes was that proposed by Imperial Chemical Industries Ltd. and called the Abbot–Cox process [14,15], in which the vat dye in its unreduced form is dispersed in Dispersol VL (0·5–1·0% solution) and circulated through the cakes prior to reduction. The Abbot–Cox process was novel in the discovery that salt can be used for exhausting undissolved pigment from the pigmentation liquors on to the cake. The salt acts as an aggregating agent for the dye pigment, gradually enlarging the size of the dye particle aggregates until they filter out inside the cake. The amount of salt required to achieve exhaustion of the pigmentation liquor varies from dye to dye, some needing no salt. It is essential that the dye liquor is exhausted before reduction, since any unexhausted pigment would, on reduction, dye on the outer surfaces of the cake and so produce unevenness.

The inventors state that the success of the process is dependent upon the evenness of distribution of the pigment through the cake before reduction and that with some dyes it is not possible to achieve this evenness, the pigment being built up on the outside or inside of the package.

After pigmentation the caustic soda and sodium hydrosulphite necessary for reduction are added to the pigmentation liquor, the Dispersol VL acting as a restraining agent for the leuco vat.

The application of vat dyes to raw stock and cotton yarn in circulating machines by the pigmentation–reduction technique was recommended by

the Calco Chemical Division of the American Cyanamid Company[16], and later some examples for package-dyeing cotton thread were given by the same firm[17].

In the Calco method, the dye is circulated through the package, caustic soda added, circulation continued, and finally the hydrosulphite added. It will be noticed that the use of salt is not advocated; however, the caustic soda added before the hydrosulphite will cause aggregation of many vat dyes in a similar way to salt, but without the same degree of control being achieved.

III— Use of Finely Divided Pigments

1. THE NEED FOR FINE PARTICLES

Bulk trials showed that the Abbot–Cox process gave greatly improved results over the normal leuco process, but that in so many cases the levelness of the dyeing left so much still to be desired that the process as it stood was applicable only in a very restricted range of dyes.

Experience with the pigmentation process showed that those dyes which needed the greatest addition of salt to the pigmentation liquor for exhaustion of the dye pigment gave the best results. Since the effect of salt could be clearly seen to be that of progressively aggregating the pigment particles, it was clear that the most important property of the vat pigment for application to viscose rayon cakes by this method was the size of the dye pigment aggregates. Since the most essential part of the process is the even distribution of the vat pigment through the viscose cake, it follows that even pigmentation will be obtained most easily from a dye where the size of pigment aggregates is such that they will first pass through the mesh of the package and subsequently aggregate slowly under the influence of salt additions, so that the particles filter out slowly and evenly in the interior mesh of filaments. In short, fine particles of pigment should give better results.

2. TESTS FOR DYE SUITABILITY IN THE PIGMENTATION PROCESS

A simple empirical test for assessing the suitability of a vat dye for the pigmentation of viscose cakes was eventually devised, the original test used being as follows— 1 g. of dye standard paste, or the equivalent quantity in other strengths, was dispersed in 0·1 g. Lissapol LS (ICI) in 40 c.c. water, and this was added to 360 c.c. water at 80°C., and allowed to stand for 5 min., when a 20 c.c. sample was removed and the rest filtered through two thicknesses of No. 1 Whatman filter paper, 7 cm. in diameter, using a vacuum filter pump. The filtration was stopped when the liquor ceased to pass freely through the filter, any liquor remaining in the funnel being carefully poured off. Skeins (1 g.) of 150/27 viscose rayon were then dyed in 20 c.c. of the original and 20 c.c. of the filtered suspension after vatting the dye in the normal manner. The following points were noted—

(1) The ease of filtration

(2) The amount of dye filtered out and its appearance

(3) The amount of dye in the filtrate compared visually with that in the original suspension

(4) The difference in depth between the two dyeings.

A good dye from the filtration test is one which filters quickly, leaving little dye on the filter paper, and consequently the filtrate contains approximately the same amount of vat pigment as the original suspension. Such a dye is said to have good filtration properties and is the most suitable for pigment dyeing processes.

Dyes possessing moderate filtration properties are those which allow only a relatively small amount of pigment to run through the filter and with which all filtration stops when 50–200 c.c. has passed. In this case the dye is intimately mixed with the fibres of the filter paper and will not dust off when dried.

Dyes possessing poor filtration properties are those with which no dye passes through the filter paper but it all remains on the surface and dusts off on drying. Any quantity of the suspension can be filtered with this class.

Measurement of the filterable particle size shows that particles which will pass through the prescribed two thicknesses of Whatman No. 1 paper have an upper limit diameter of about 2–3 μ. Repeated attempts at machine dyeing with pigments of various degrees of dispersion showed that this dimension was about right for use in bulk practice. Particle sizes above this value have given unsatisfactory results.

Now the ability of a pigment to pass this dispersion test tells us only that it can be uniformly distributed, by salt aggregation, throughout the cake. The degree of levelness resulting from ultimate conversion into the leuco form and a more or less prolonged process of dyeing from a hot alkaline bath will further depend upon the diffusion or migration properties of the substantive leuco dye proper. Whilst dye is migrating from the cake interstices to the inside of the fibre, reduced and partly reduced pigment is being washed away from the fibre surfaces by solution in, and movement with, the circulating reducing liquor. Therefore, after pigmentation, the most level results will be obtained, in the shortest time, by those dyes with the highest migrating speeds.

To ensure the selection of suitable dyes, therefore, the results of filtration tests are considered alongside those of a migration test. Further, since our uniformly pigmented cake has still to undergo dyeing proper, and adsorbed pigment, as outlined above, dissolves and tends to redistribute as dye in solution, which dye has a high rate of readsorption (though not of diffusion) in its leuco form, the degree to which a dye may be restrained is still an important property. Hence, in classifying dyes for suitability for cake dyeing, a test is made for this property.

The migration test, which measures redistribution of the dye, is described in the Vat Dyes Report[5], and consists in treating a dyed skein with a white skein of equal weight in a blank dyebath containing the same amount of caustic soda and sodium hydrosulphite as used in the original dyeing

and noting the degree of migration from the dyed to the white skein for a given time, temperature, and liquor ratio. The conditions can be altered to suit the required conditions of dyeing.

The test for the degree of restraining is carried out by dyeing a number of skeins in separate baths with the dye under test vatted in the usual way; in one case no restraining agent is added, and in the others various amounts of the restraining agent under test. The tests are carried out under the desired conditions of temperature, liquor ratio, and time. The degree of restraining is assessed by the difference in depth between the various dyeings.

Speke [10] gives the restraining action of Dispersol VL on the various Caledon (ICI) dyes, using a test similar to that regularly applied by the present author to all new dyes with cellulose sulphite waste liquor. The tests can be combined by the use of the strike–migration or strike–levelling test [2–5], various amounts of the restraining agent being added to test liquors. The effect on the strike of the restraining agent can also be studied by this test.

Dispersol VL gives a degree of retardation which is successful under the conditions of pigmentation dyeing of viscose rayon cakes and is in use in our present practice. Experiments are proceeding to ascertain whether this agent or cellulose sulphite waste liquor is more economical to use, the latter being satisfactorily employed in the leuco vat dyeing of viscose rayon in hank.

Experience in the laboratory and the dyehouse has shown that commercial dyes may be roughly divided, according to their suitability for dyeing, into the classes shown diagrammatically in Table I.

TABLE I

Filtration	Migration	Degree of Restraining	Class	
Good	Good	Strong	1	A
		Weak	2	
	Poor	Strong	2	
		Weak	3	
Moderate	Good	Strong	4	B
		Weak	5	
	Poor	Strong	5	
		Weak	6	
Poor	Good	Strong	7	C
		Weak	8	
	Poor	Strong	8	
		Weak	9	

As usual with such a classification, borderline cases exist between the various classes, and it is difficult to differentiate between A3 and B4. Dyes of class A1 and A2, however, are ideal for the purpose, and the remaining classes are progressively more difficult to apply. Dyes in Class C give very poor results.

3. DYES CLASSIFIED ACCORDING TO TEST

A large number of vat dyes were tested, and the results show that dyes possess wide differences in filtration properties. Fig. 1 shows examples of dyes belonging to various classes, while Fig. 2 indicates that the brands of various manufacturers or successive deliveries of the same dye possess different filtration properties. Very few dyes tested have fulfilled the ideal conditions.

4. GRINDING OF COMMERCIAL DYES

Since it was desirable that the dye pigment gave good filtration, various methods of reducing dye aggregate size were tried and included passing the dye paste through (1) a homogeniser, in which the dye paste passed between two moving steel plates, and (2) an emulsifier, in which the dye paste is forced through a fine jet under pressure. These methods did not effect sufficient reduction in particle size.

It is well known that ball and pebble mills are used for breaking down the pigment aggregates of a large number of solid organic substances, and the use of such mills is described by Mills [18] and Fischer [19]. Attempts to reduce the pigment aggregates of vat dyes in common use in the dyehouse were made on a laboratory-size ball-mill and proved successful; works dyeings made from dyes reground in the laboratory in 1-lb. lots immediately gave results vastly superior in levelness to the dyeings made from the original dye paste.

As a result of these trials all commercial vat dyes which do not pass filtration tests are reground in a pebble mill before use in the Droylsden dyehouse. The time taken for regrinding varies from dye to dye, some being satisfactory after two days whilst some require seven days before passing the simple test described. They then fall into Class A. No instance has been observed of re-aggregation of the reground dye pigment; samples kept in the laboratory over 18 months are still satisfactory in the filtration test.

The filtration properties of various dyes before and after regrinding are illustrated in Fig. 3, and photomicrographs of the pigments are shown in Fig. 4.

The dyeing procedure has had to be modified, since the reground pigments require the addition of salt to make a 1–2% solution in the pigmentation liquor in the majority of cases, before exhaustion of the dye pigment is complete. This concentration of salt, if carried over to the reduction stage, would frequently increase the strike and decrease the levelling powers of the leuco dye. The pigmentation step is carried out in 0·25 part of Lissapol LS per 1000, and the reduction is carried out in a fresh bath containing the necessary quantities of caustic soda, hydrosulphite, and Dispersol VL, since to retain the pigmentation bath is to introduce too much salt for control of leuco dye exhaustion.

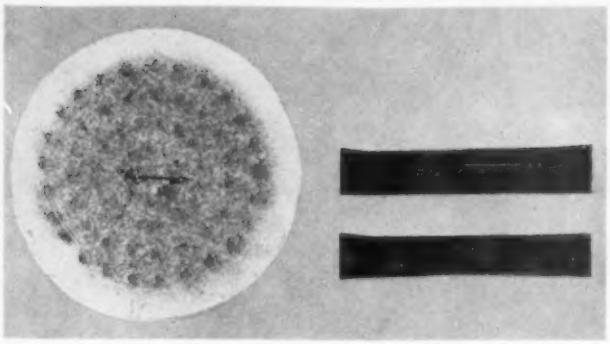
5. EXPERIENCE OF MODIFIED PIGMENT PROCESS

The use of finely ground vat dyes applied by the method described represents, in terms of dyehouse practice, a great advance over the leuco dyeing method or the original Abbot–Cox process. It has enabled the production for the first time of a satisfactory range of shades vat-dyed on rayon cakes to commercially acceptable standards of levelness of shade and processing condition of the dyed yarns. The modified process does not give satisfactory results with unground dyes.

IV—Dyeing Practice

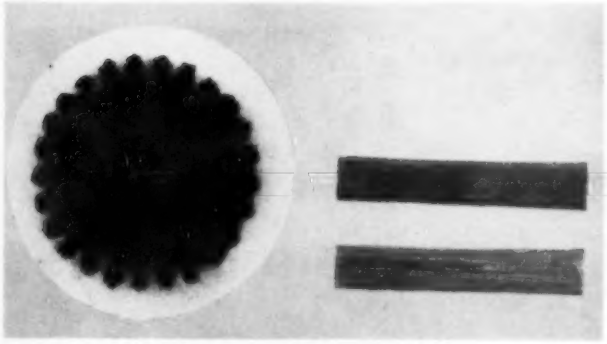
1. DRESSING

The dyeing of viscose rayon cakes is generally carried out in the Droylsden dyehouse on two types of machines, the Longclose and the Courtaulds



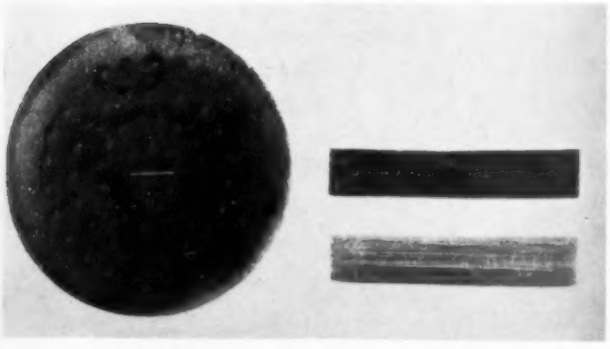
(i) 380 c.c. filtered (all)

(A) Commercial Dye showing Good Filtration Properties



(i) 85 c.c. filtered

(B) Commercial Dye showing Moderate Filtration Properties

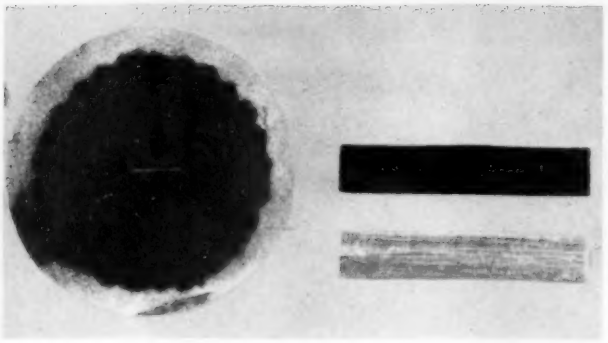


(i) 190 c.c. filtered

(C) Commercial Dye showing Poor Filtration Properties

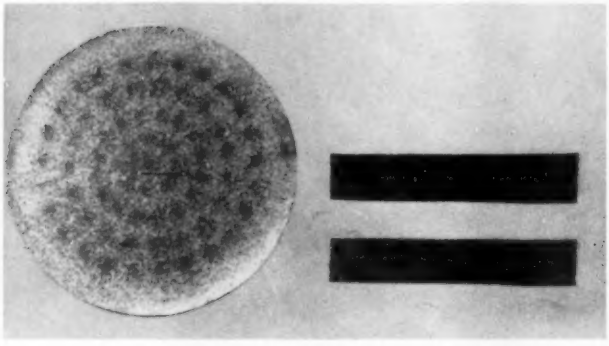
(ii) Original suspension } 1 g. 150/27 Viscose rayon dyed in 20 c.c.
(iii) Filtered suspension } suspension, vatted in normal way at 60°C.

FIG. 1—Filtration Test of Vat Dyes showing Different Filtration Properties



(i) 140 c.c. filtered

(A) Dye I as delivered

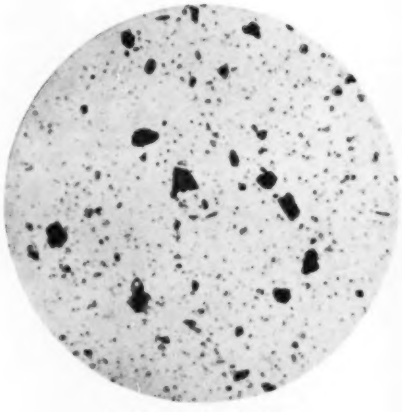


(i) 380 c.c. filtered (all)

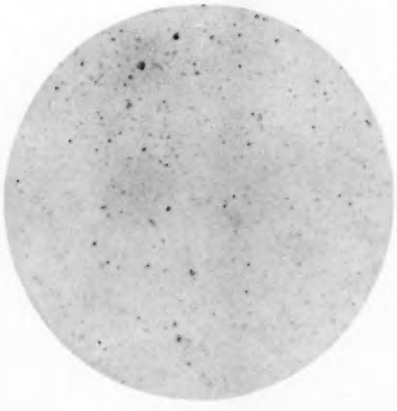
(B) Dye I reground

(ii) Original suspension
(iii) Filtered suspension

FIG. 3— Filtration T



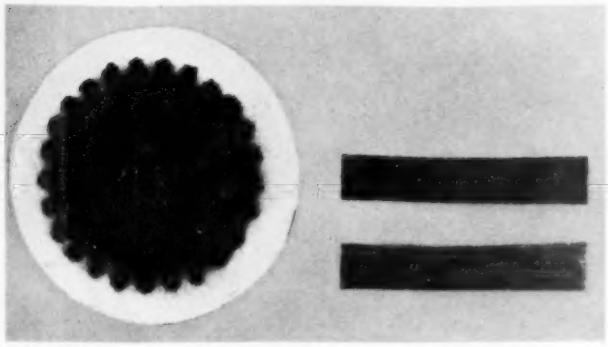
As delivered before Regrinding



After Regrinding

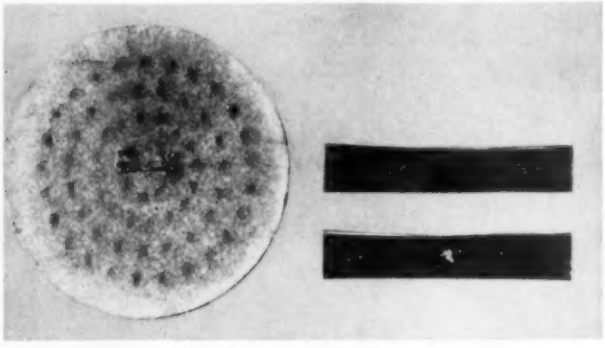
Dye I

FIG. 4— Photomicrographs



(i) 85 c.c. filtered

(C) Dye II as delivered

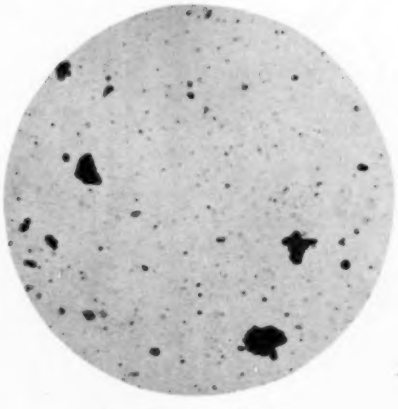


(i) 380 c.c. filtered (all)

(D) Dye II reground

Viscose rayon dyed in 20 c.c.
n, vatted in normal way at 60°C.

Effect of Regrinding

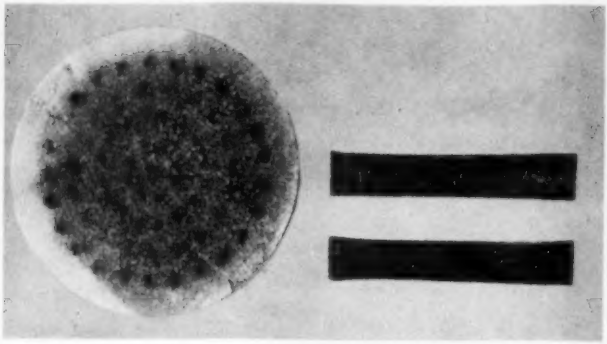


As delivered before Regrinding

After Regrinding

Dye II

owing Effect of Regrinding

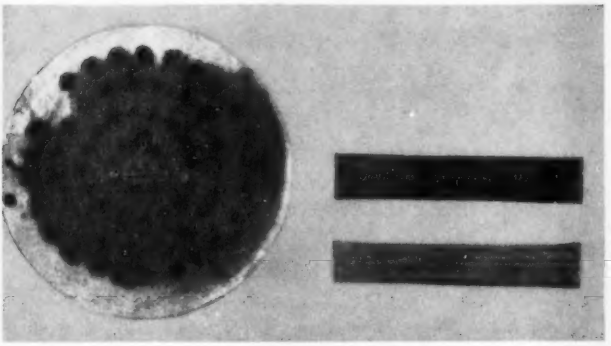


(ii)

(iii)

(i) 380 c.c. filtered (all)

(A) Dye showing Good Filtration Properties

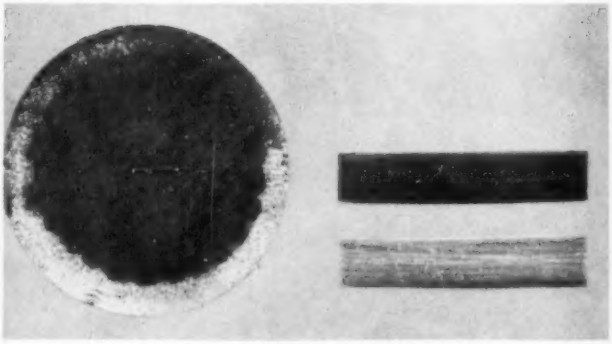


(ii)

(iii)

(i) 135 c.c. filtered

(B) Dye showing Moderate Filtration Properties



(ii)

(iii)

(i) 100 c.c. filtered

(C) Dye showing Poor Filtration Properties

(ii) Original suspension
(iii) Filtered suspension

} 1 g. 150/27 Viscose rayon dyed in 20 c.c. suspension, vatted in normal way at 60° C.

FIG. 2— Filtration Tests of Various Manufacturers' Brands of the Same Dye showing Different Filtration Properties

dyeing machines, both of which have been described by Whittaker [1,20] as used for this purpose. The Courtaulds machine lends itself particularly well to vat pigment dyeing, and the method of dressing it may be described.

The viscose rayon cakes are dressed on stainless steel or plastic cake holders of such a size that the cakes are a good fit on the holders. Cakes which are held slackly on the holders will give inferior results owing to uneven flow through the cake; also there is a greater danger of cake collapse with a badly fitting cake. This method of dressing is different from that described by Whittaker for the Courtaulds machine, where the cakes are dressed on fluted tubes [20]. The holders are so made that the top end of one interlocks with the bottom end of the next, and thus each cake is completely separated. The spacing between the flanges of separate holders is such that the cakes are held rigid between solid supports, and so danger of collapse is much less than when the cakes are dressed cake to cake on tubes.

The cakes are dressed in a horizontal position, the end holder being fixed with a fitted flat lid and the whole fastened by an end nut. On the Courtaulds machine the cakes are wetted out in boiling water until all the air has been displaced, the frame is then lifted off the bottom of the tank, the pump started, the frame gradually lowered into position, and the liquor circulated for 10 min. This procedure is adopted to avoid the sudden build-up of pressure on the cake wall, which would tend to cause collapse. The frame is removed from the liquor, and the pigmentation bath prepared. In the Longclose machine, the cakes are dressed on the holders and stacked vertically in the manner described by Whittaker [1]. The cakes are wetted out before dyeing.

2. DYEING

The dye of correct particle aggregate size is mixed in small volume with Lissapol LS in solution (0·25 part per 1000 calculated on the total dye liquor). This is added to the dye liquor at 82°C. (180°F.), the cakes are entered, and the pigmentation liquor is circulated for 30 min. The salt in solution is then drip-fed into the pigmentation liquor over one hour, when the pigment should be completely exhausted from the liquor. The amount of salt required varies according to the amount of dye present, no salt being required for very thin shades and salt to make a 2·0% solution in the total dye liquor for heavy shades. Very few shades require no salt to exhaust the pigment. The frame is now raised out of the liquor, the exhausted liquor run off, and a fresh bath prepared for the reduction stage with the necessary amounts of caustic soda, sodium hydrosulphite, Dispersol VL, and tannic acid.

(1) Caustic Soda Addition—(*a*) *Strong-alkali Dyes*—1·75 c.c. caustic soda (70°Tw.) per 100 c.c. liquor. With Caledon Blue RC the amount of caustic soda used is increased to 2·5 c.c. per 100 c.c. owing to the poor solubility of the leuco dye–Dispersol VL complex.

(*b*) *Weak-alkali Dyes*—1·0 c.c. caustic soda (70°Tw.) per 100 c.c. liquor.

Since the dyeing is generally carried out at higher temperatures (80°C.) than usual, extra hydrosulphite is necessary owing to the greater rate of decomposition at this temperature and consequent neutralisation of caustic soda by the decomposition products of the hydrosulphite. This could result, in the case of weak-alkali dyes, in the liquor becoming acid, and with the strong-alkali dyes the alkali concentration might become less than necessary for the best dyeing conditions. The alkali concentration of the dye liquor is therefore increased.

(2) Sodium Hydrosulphite—10·0% on the weight of material, one-half of this being added at the start of the dyeing, the rest being added during the dyeing operation.

(3) Dispersol VL—10% on the weight of material.

(4) Tannic Acid—0·4 part per 1000. This is added to assist in the prevention of degradation of the cellulose during dyeing. The tannic acid is preferentially oxidised instead of the viscose rayon [21].

Time and Temperature of Dyeing

(*a*) Strong-alkali Dyes—Strong-alkali dyes are dyed for 2 hr. at 80°C. (176°F.), except those dyes which are decomposed at that temperature, e.g. Caledon Blue RC, Purple 4R, and Brilliant Orange 4R types, which lose halogen at elevated temperatures. These dyes are dyed at 60–65°C. (140–150°F.).

(*b*) Weak-alkali Dyes—(*i*) *Hot-dyeing Type*—These are dyed for 2 hr. at 80°C. (176°F.).

(*ii*) *Cold-dyeing Type*—These are dyed for 1 hr. at 60°C. (140°F.), and then cooled to 38°C. (100°F.) over 1 hr. before oxidation.

(*iii*) *Mixtures of Hot- and Cold-dyeing Types*—These are dyed for 1 hr. at 80°C. (176°F.) and then cooled to 38°C. (100°F.) over 1 hr. before oxidation.

During dyeing the following details are important—

(1) Since the dyeing tank of the Courtaulds machine presents a comparatively large surface area to the atmosphere, covers are placed over the tank during dyeing to prevent light from falling on the leuco dye solution, as many leuco compounds are very sensitive to the action of light.

(2) The exposure of the cakes to light during dyeing and before oxidation must be avoided as far as possible, since with certain dyes degradation of the viscose rayon on the outside of the cake will occur. An example of this is given in Table II,

Table II

(Single-thread wet-strength tests on 150/27 viscose rayon cakes dyed with Caledon Golden Orange 3G (ICI) and exposed to light between dyeing and oxidation)

B ... Breaking load (g.)
E ... Extension at break (%)

Position in cake...	Outside		Middle		Inside	
	B	*E*	*B*	*E*	*B*	*E*
White undyed yarn	139·0	19·2	141·0	18·8	141·0	21·3
Dyed cake exposed to light between dyeing and oxidation	118·0	11·4	131·0	16·6	134·0	17·4
Dyed cake not exposed to light between dyeing and oxidation	132·0	16·5	134·0	16·9	138·0	17·3

which shows the greatly increased loss in wet strength of the viscose rayon on the outside of the cake.

Scholefield and Patel[22] describe the tendering of cotton dyed with certain vat dyes on exposure of the leuco-dyed material to light. Similar work has been carried out on viscose rayon (150/27), and some results are given in Table III.

TABLE III

(Single-thread wet-strength tests as in Table II)

Caledon (ICI) Dye	Air Oxidation in the—	*B*	*E*
White undyed yarn	—	140·4	22·0
Golden Orange 3G ...	Light	99·0	11·9
	Dark	146·6	20·0
Golden Orange G ...	Light	74·7	9·92
	Dark	140·7	18·0
Yellow 5GK ...	Light	119·4	13·8
	Dark	145·4	30·0
Yellow 5G ...	Light	71·5	9·32
	Dark	139·0	18·5
Red 3BS	Light	97·8	4·0
	Dark	141·8	20·93
Brown R	Light	136·6	17·3
	Dark	145·2	20·3
Blue RC	Light	144·2	18·6
	Dark	141·7	18·0

(3) The dyeing temperature must not exceed 80°c. (176°F.). The effect of a rise of 5°c. over the maximum dyeing temperature on yarn strength is shown in Table IV. These results were obtained in a works bulk dyeing.

TABLE IV

(Single-thread wet-strength tests as in Table II on 150/27 viscose rayon cakes dyed with 0·6% Caledon Brown RS paste and 2·4% Caledon Brown GS paste)

	B	*E*
White undyed yarn ...	139·0	19·3
Dyed 2 hr. at 85°c. ...	94·0	9·1
Dyed 2 hr. at 80°c. ...	131·0	16·4

3. OXIDATION

On completion of the dyeing, oxidation is carried out by circulation of sodium hypochlorite, the method varying with the type of dye used—

(*a*) STRONG-ALKALI types are first washed in cold water and then oxidised with 0·67 c.c. chemic (15% available chlorine) per 100 c.c. Dyes of the Caledon Yellow GN type are treated in 0·3 part of sodium bicarbonate per 100 before chemicking; this reduces the alkalinity of the liquor left in the cake.

(*b*) WEAK-ALKALI (HOT-DYEING) types are washed in 2% salt solution and then oxidised with chemic (as (*a*)) in 2% salt solution.

(*c*) WEAK-ALKALI (COLD-DYEING) types, the leuco compounds of which are very soluble, are oxidised, without a preliminary wash, in 2% salt solution containing 1·3 c.c. chemic per 100 c.c.

When oxidised, the cakes are given two cold washes, then one hot wash, and are finally soaped at the boil with 0·2% soap solution, 0·0125% Lissapol LS solution, 0·025% Calgon T solution.

V—Conclusions

The use of reground vat dye pigments has greatly increased the range of shades that can now be dyed on viscose rayon cakes of normal deniers on the Longclose and Courtaulds dyeing machines. There are still many problems to be solved, and two may be mentioned—

(1) The use of a reduced amount of restraining agent with heavy shades of strongly restrained vat dyes, thus allowing heavy shades to be produced more economically.

(2) The use of mixtures of strong- and weak-alkali dyes in one bath.

It has been stressed many times that, in the dyeing of viscose rayon by normal leuco methods, strong- and weak-alkali dyes should not be used together in mixtures owing to the wide differences in rate of strike, rate of diffusion, and alkali concentration needed in dyeing; but it might well be that these do not have the same bearing on the dyeing when the dye pigment has been distributed evenly through the cake. Work is in progress on these problems and, indeed, shows considerable promise.

Finally, the principle of vat pigmentation is being extended experimentally to other types of circulatory dyeing, e.g. of rayon staple tops, loose Fibro, and yarns, including blends with other fibres.

* * *

Since the date of preparation of this paper, the technique of pigmentation–reduction using finely ground vat dyes has been applied to viscose rayon staple in sliver and top form. With the rapidly increasing use of viscose rayon staple in the worsted trade the importance of methods for slubbing dyeing with vat dyes is self-evident. This development has been fully described by Boulton[23] in a lecture to the West Riding Section of the Society.

* * *

The author desires to thank the Directors of Messrs. Courtaulds Ltd. for permission to publish this paper, Mr. E. R. Lambie for continuous dye-house co-operation, and Mr. J. Boulton for advice and help.

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(*Received 17th March 1951*)

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[21] Whittaker and Wilcock, *op. cit.*, p. 44.
[22] Scholefield and Patel, J.S.D.C., **44**, 271 (1928).
[23] Boulton, *ibid.*, **67**, 401 (Nov. 1951).

CORRESPONDENCE

The Editor does not hold himself responsible for opinions expressed by correspondents

Development of the Society's Light Fastness Standards

The Editor

SIR,

In so far as the train of thought pursued in the article by the Fastness Tests Co-ordinating Committee [1] may be summarised in the sentence: "It is clear, therefore, that the German and British standards were worked out simultaneously", there is no objection to raise. I refer in this respect to an article by Ris [2] in March 1948, in which he writes: "Independently of this work [of the D.E.K.] the Society of Dyers and Colourists has followed similar lines in England since 1930. As the first result of their systematic work on a broad basis, a red and a blue scale were officially issued." And in an unpublished report of Hanns Rein (formerly I.G., Mainkur), who played a major part in the work in question, there is at one point the statement: "My remarks [regarding the work on the production of a blue scale] would not be complete without giving credit to the endeavours of other countries. The S.D.C. has for years been devoting the greatest attention to the assessment of light fastness. The broad basis of the series of tests they have conducted has also clearly shown that only dyeings on wool are acceptable as a standard. Independently of our work, they have also established a blue scale. A red scale will also certainly be established."

The extracts from recent papers quoted at the beginning of the F.T.C.C. article [1] were certainly dictated by no spirit of ignoring the work of the S.D.C. Apart from the fact that it is always rather vague to quote single sentences out of context, they have been included in the papers cited in order to furnish documentary proof that efforts were also being made in the same direction in questions of the standardisation of light fastness in other European countries, particularly in England. Is this reasoning so very far wrong if one disregards the temporal concurrence of the development work of the S.D.C. and the D.E.K. and considers only the purely formal side of the final composition of the English and German light fastness scales?

1932— First official publication [3] regarding one eight-step blue scale on the part of the Germans
1934— First publication [4] of a seven-step red and blue scale
1939— Final establishment of the Types of the D.E.K. standards which are still valid to-day
1940— Establishment of the S.D.C. standards still valid to-day (as the F.T.C.C. says at the end of its paper [1], ". . . in order to come into line with the D.E.K."), which with minor discrepancies agree very well with one another, and are also, as Ris showed, similar in their grading on exposure to light.

The more recent statements of Ris, Zuber, and myself quoted by the F.T.C.C. can be understood in the light of this and the realisation of the fact which emerges that in the leading European dye-producing countries a large measure of agreement exists in the matter of light fastness standardisation. Thus they were not made as claims to priority but to show that, at least as far as Europe is concerned, nothing stands in the way of a unification of the test methods for the assessment of fastness to light.

For the sake of a clear picture with respect to the independence and simultaneity of the development work in England and on the Continent, I may be permitted to take up several further remarks of the F.T.C.C. The main point in this connection is the influence of moisture on the result of an exposure to light, and in particular the realisation that dyeings on wool are more independent of climatic conditions than dyeings on cotton.

The fact that moisture plays a rôle in exposure to light really dates back to Chevreul, who showed that dyeings exposed to light in a vacuum fade almost imperceptibly or not at all. This is first recorded *inter alia* in the fundamental works of Gebhard [5]. It was also Gebhard who first drew attention, in the same paper, to the importance of the substrate for the result of the exposure to light. Particular reference is made by the same author in this connection in his third paper on "The Effect of Light on Dye Systems" [6], in which he deals with the increased rate at which the dye–cotton system breaks down as compared with the dye–wool system.

I am going into these matters because the remarks of the F.T.C.C. might create the impression that the simultaneous and independent work of the D.E.K., in the course of which the earlier results outlined above were likewise confirmed and which led to the blue wool scale, was influenced by the work of the S.D.C.

Dr. P. W. Cunliffe [7] referred to "The [British] work of 1928 and 1929 having shown that silk and wool are the best fibres to use for the standard dyeings, . . ." and in 1934 it was stated that "It is a significant fact that the proposals made at the time [i.e. 1930] by the Society's Fastness Committee have been subsequently used by prominent German and American organisations interested in the subject [meaning fastness committees]."

If the first blue standard scale was published by the former I.G. in 1932, the F.T.C.C. will be best

able to judge from knowledge of the difficulties of such tedious work that years of intensive tests must have preceded this publication before serviceable and repeatedly checked results were available. As a matter of fact, we began our work early in 1927, and a scientific summary of the most important points appeared at the beginning of 1933 as the second communication on "Light Fastness and its Standardisation". A fuller account of the results obtained was given by H. Rein in a lecture in Würzburg on 9th June 1933 [9].

All the work on the production of suitable light fastness standards was carried out within the former I.G., and only the final draft was submitted to the D.E.K. for approval. It may thus be understandable that in 1930 Krais should still defend the different standards in use until then. The work of the I.G. in this field was certainly unknown to him at that time.

Unfortunately, I no longer have available evidence concerning the details of the negotiations carried on in 1939 between Krais and the S.D.C. and the A.A.T.C.C., as his notes were destroyed during the war. I would like to point out in this connection, however, that at that time an agreement already existed with Switzerland regarding the joint use of the blue wool scale, which means that Krais also had to negotiate at the same time in their name.

Hence, when the F.T.C.C. itself remarks that the development was carried out simultaneously and independently in England and on the Continent, I should like to emphasise again, in conclusion, that in this connection we can entirely agree.

All in all, however, at the present time it seems to me that the question of priority does not stand so much to the fore as the fact of being on more or less the same ground. This especially in view of the fact that we can regard the February meeting in Basle as a good start to international co-operation.

Yours faithfully

P. RABE

FARBENFABRIKEN BAYER
LEVERKUSEN
GERMANY

16th June 1951

[1] Fastness Tests Co-ordinating Committee, J.S.D.C., **67**, 188 (May 1951).
[2] Ris, H., *Textil-Rund.*, **3**, 85 (1948).
[3] *Melliand Textilber.*, **13**, 539 (1932).
[4] "Report of the Society of Dyers and Colourists on the work of its Fastness Committee in fixing Standards for Light, Perspiration and Washing" (Bradford 1934).
[5] Gebhard, *Z. angewandte Chem.*, **22**, 1892 (1909).
[6] Idem, *ibid.*, **23**, 823 (1910).
[7] Cunliffe, P. W., J.S.D.C., **47**, 8 (1931).
[8] Fastness Tests Report [4], p. 12.
[9] Rein, H., *Z. angewandte Chem.*, **47**, 157 (1934).

Comparison of Fading Rates by means of the Society's Light Fastness Standards (B.S. 1006)

The Editor

SIR,

The quantitative study of the fading of dyes on the fibre has been largely neglected owing to the difficulty of measurement of the rate of actual destruction of dye. Extraction of unchanged dye from the faded fibre is often impracticable, and reflectance measurements require elaborate apparatus and are difficult to interpret, though they have been used for this purpose, on wool, by Kienle, Stearns, and van der Meulen [1]. The difficulty does not, of course, arise when transparent substrates are used, and Kienle *et al.* and Atherton and Seltzer [2] measured fading rates spectrophotometrically on gelatin and cellulose acetate films respectively. Simple benzeneazonaphthalene dyes were used by these authors, and rate data were plotted against the Hammett σ values* of the substituent groups in the nucleus of the diazo component. Linear curves were obtained.

We have found that relative fading rates of dyes on opaque surfaces may be compared quantitatively in a simple manner, using the Society's light fastness standards [3].

The criterion we have adopted is the first just perceptible change. It may be assumed that the fade represents in each case the destruction of approximately the same fraction a (probably of the order of 5%) of total dye present.

It was originally intended that each light fastness standard should fade twice as fast as the one next higher [4]. This ideal was not attained, and in particular those above grade 6 are not so uniformly spaced. Our tests are mainly based on samples graded from 2 to 5–6, and, as a first approximation, we assume that the standards between these limits are uniformly spaced. Thus a, the fixed proportion of dye faded, should be destroyed in a time $2^{n-1}t$, or $2^n t/2$, where t is the time for Standard 1 to show a just perceptible fade and n is the grade number of the pattern under test.

It follows that in the time t a proportion $2a/2^n$ of the original dye is destroyed.

The reaction rate of the fading of any dye may be measured by the quantity of that dye destroyed in a given time, say t, and it may thus be expressed as $2ak/2^n$, where k is a constant. The rate is thus inversely proportional to the antilogarithm of the grade number. Hence, if the grade numbers of a substituted dye and the corresponding unsubstituted product are n and n_0 respectively, then the ratio of their rates of destruction is $n_0 - n$. This ratio may be plotted against the corresponding σ values for the substituent groups, to show the influence of substituents in a series of dyes upon their fading rates.

We have found that such a plot for a series of simple azo dyes on different substrates is linear, as was observed for the plots of reaction rates by the above-mentioned workers. Plots of data from single patterns of each dye show somewhat wide scatter about the best straight line, as would be expected in view of the large number of possible sources of error in visual light fastness assessment, but mean values of observations upon several patterns of each dye give improved plots. Fig. 1 shows the data for a series of dyes prepared by coupling diazotised aniline and some of its *m*- and

* A quantitative measure of the electrophilic nature of *meta* and *para* substituent groups in a benzene nucleus.

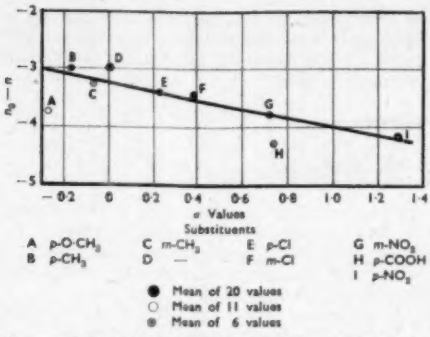

FIG. 1— Fading Rate of Substituted Benzeneazo-β-naphthol Dyes

p-substituted derivatives with β-naphthol and certain derivatives, exposed to light on cellulose and porous porcelain tile. The individual grade numbers covered the range from 1–2 up to 5–6.

Further details will be published later.

Yours faithfully

N. F. DESAI
C. H. GILES

COLOUR CHEMISTRY RESEARCH LABORATORY
DEPARTMENT OF TECHNICAL CHEMISTRY
ROYAL TECHNICAL COLLEGE
GLASGOW C.1

21st August 1951

[1] Kienle, Stearns, and van der Meulen, *J. Physical Chem.*, **50**, 363 (1946).
[2] Atherton and Seltzer, J.S.D.C., **65**, 629 (1949).
[3] *B.S.* 1006 (British Standards Institution).
[4] "Report of the Society of Dyers and Colourists on the work of its Fastness Committee in Fixing Standards for Light, Perspiration, and Washing" (Bradford: the Society 1934); J.S.D.C., **56**, 273 (1940).

Notes

Proceedings of the Council

At a meeting of the Council, held at the offices of the Society, 32–34 Piccadilly, Bradford, on 8th August 1951, the proceedings included the following items of interest—

A.C.I.T.— The President reported that he would be unable to accept the invitation to attend the Annual Congress of the Association des Chimistes de l'Industrie Textile, to be held in Paris on 28–30th September 1951. Arrangements have since been made for the Society to be represented by Mr. J. Boulton and Mr. E. A. Swift.

A.I.C.T.C.— It was resolved that the Society's good wishes for the Associazione Italiana di Chimica Tessile e Coloristica be formally conveyed to Dr. L. Sessa, Chairman of the Italian Association.

HALF-YEARLY ACCOUNTS— A draft statement of accounts and balance sheet for the half-year ending 30th June 1951 was approved.

FASTNESS TESTS CO-ORDINATING COMMITTEE— It was reported that Mr. K. MCLAREN had undertaken the duties of Honorary Secretary of this Committee.

MEMBERSHIP— Eleven applications for ordinary membership and two for junior membership were approved. Mr. E. St. J. Blakeley and Mr. J. W. Young were readmitted to membership.

NINTH MERCER LECTURE— It was reported that the Mercer Lecture Committee recommended that the 1952 Lecture should be held in the Midlands area and should deal with the shrinkproofing of wool and wool-mixture materials. Dr. F. C. Wood has since accepted an invitation to deliver this Lecture.

Meetings of Council and Committees September

Council— 12th
Finance— 12th
Publications— 25th
Colour Index Editorial Panel— 3rd
Bye-laws— 5th

Deaths

We regret to report the loss by death of Mr. J. Baddiley, Mr. W. H. Clarke, and Mr. S. G. Mills.

A.G.M. and Dinner 1952

Arrangements have been made to hold the 1952 Annual General Meeting and Dinner on Friday, March 28th, at the Queens Hotel, Leeds.

Congrès International de la Recherche Scientifique appliquée à l'Industrie Textile

The International Congress of Textile Research organised by the Institut Textile de France was held in the buildings of the International Textile Exhibition at Lille on 8–9th May 1951. Of the hundred members who attended, a substantial proportion were from overseas, thereby stressing the international character of the meeting. The conference was divided into four sections according to the subject-matter of the papers read— (*a*) General Questions, referring to such matters as the use of the tristimulus system of colour measurement, microscopy. (*b*) Cellulose and Synthetic Fibres. Here, Great Britain was represented by Dr. D. Traill, who gave a discussion on the preparation of synthetic fibres. (*c*) Wool and Vegetable Fibres. This was the most active section, and included papers on the dissolution of wool and cystine estimations by Prof. J. B. Speakman together with contributions from the Wool Industries Research Association and the International Wool Secretariat. (*d*) Processing and Finishing. This section was rather mixed, containing papers from W. J. Marshall and R. H. Peters of the Society on dye properties, C. S. Whewell from Leeds University on raising, and J. M. Preston from Manchester College of Technology on drying of fibres. Although Great Britain was well represented at the congress, the majority of the papers came from French institutions, producing some very interesting data and results.

Overshadowing the conference, however, was the extremely kind hospitality shown by the Institut and some of the local residents, on whom several members were billeted. The conference was entertained by the Chambre de Commerce de Roubaix for lunch and a visit to their laboratories; and by the Institut to both lunch and an excellent symphony concert. R. H. PETERS

New Books and Publications

Papers delivered at the Second Canadian Textile Seminar

(i) Preprint of Papers. Pp. viii + 201. Sept. 1950.
(ii) Supplement, containing additional text and report of discussions. Pp. 107. [Multilitho.] May 1951.
Montreal: Textile Technical Federation of Canada. Price, $5.00.

In a notice appearing in this *Journal* (**65**, 289 (1949)) upon the papers given at the First Canadian Textile Seminar, the Textile Technical Federation of Canada was congratulated upon its enterprise and assured of the good wishes of this Society. These congratulations must be renewed for the present effort. It would be very pleasant for members of this Society to think that the good wishes have proved to be an encouragement to the Federation in this, their second and decidedly more mature effort.

The word seminar is not much used in this country, and, in judging the range, style, and quality of the papers, the implication of deliberate educative value (the seed-planting for a harvest of better technical practice) must not be lost sight of. Thus, in aim at any rate, this gathering is more closely allied to the summer schools and refresher courses of our own technical societies than to the symposia and conferences in which efforts are made to plumb the depths of a more narrowly restricted subject matter.

As at the 1948 seminar, the range is wide, from factory organisation, through the various stages of textile manufacture and processing, to testing and engineering. In general, the papers give synoptic treatments, with particular emphasis on new technical developments. A few are indeed so short and summary that, on reading, it seemed probable that they would be extended verbally at the actual presentation, and this is confirmed by the length of the discussion which follows. The balance of subjects is a decided improvement over that of 1948, and it is good to see that, while many papers come from the Canadian experts, visitors from outside the country are well represented, including Messrs. Ashner, White, Strang, von Bergen, Dreby, and Valko from the United States, and Messrs. Ashton, McFarlane, Wilcock, and Douglas from this country.

To comment upon each paper contributed is beyond the possibility of this review, so that, with no invidious intention, a few may be singled out for brief notice. Two full papers, discussing the still recondite details of the action of the card, A. Varga dealing with worker–stripper cards, and P. M. Strang with revolving flat cards, reflect the wide interest that is being taken in this remarkable and obscure micromechanical process. The influence of the manufactured fibres upon all branches of textile production receives constant recognition, whether it be in the production of wool–rayon staple fibre yarns (H. Ashton), in sizing (G. LaPiana), in the setting (F. S. Kenny and R. E. Fryer) and dyeing (G. T. Douglas) of nylon, the dyeing and finishing of cellulose acetate warp-knitted fabrics (T. H. Hilliard), or in other context. Among the general introductory treatments, that of M. L. Staples, containing no novel material but relating many of the chemical properties of fibres with the molecular constitution of the fibre substance, is mentioned as a competent and interesting summary. R. A. McFarlane and C. C. Wilcock review developments in textile processing machinery with a thoroughness which we have come to associate with them. An attempt by E. I. Valko to relate finishing processes (considered in the wider sense of *Veredelung*) to underlying scientific principles reads less happily. A most interesting inclusion in the programme is a short outline by M. H. Thomas of the handling and use of radioactive isotopes. Not many examples of specific textile application are given— not so very many can exist— but there is the obvious and praiseworthy intention of encouraging their future use in textile investigation.

The proceedings make clear how strongly the Canadian industry can be influenced by relations with both Great Britain and the United States. Many of Canada's technicians are British born and trained, while there are the close geographical and economic ties with the United States. As her own industries develop, she has thus the opportunity of producing a happy and characteristic synthesis of the long tradition of invention in the one country with the special genius of the other for industrial adaptation. H. A. TURNER

Technique of Organic Chemistry
Volume IV
Distillation

Edited by A. Weissberger. Pp. xxvii + 668. New York and London: Interscience Publishers. 1951. Price, $14.00 or 112s. 0d.

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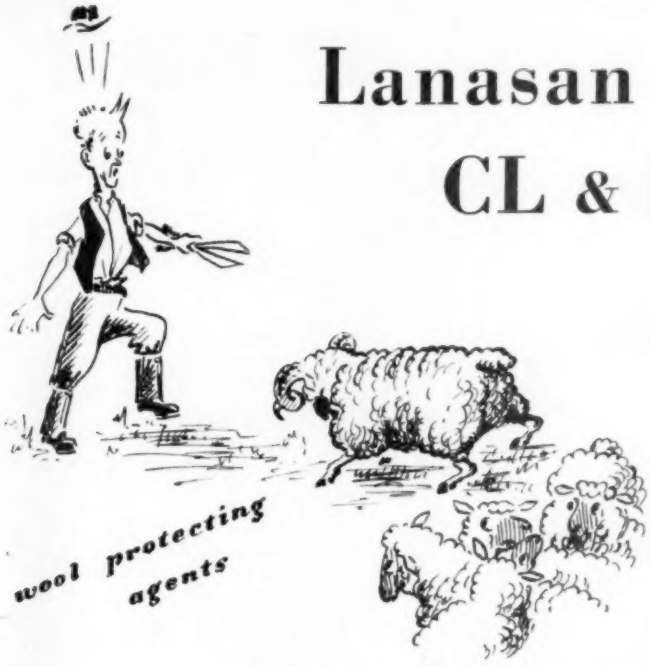

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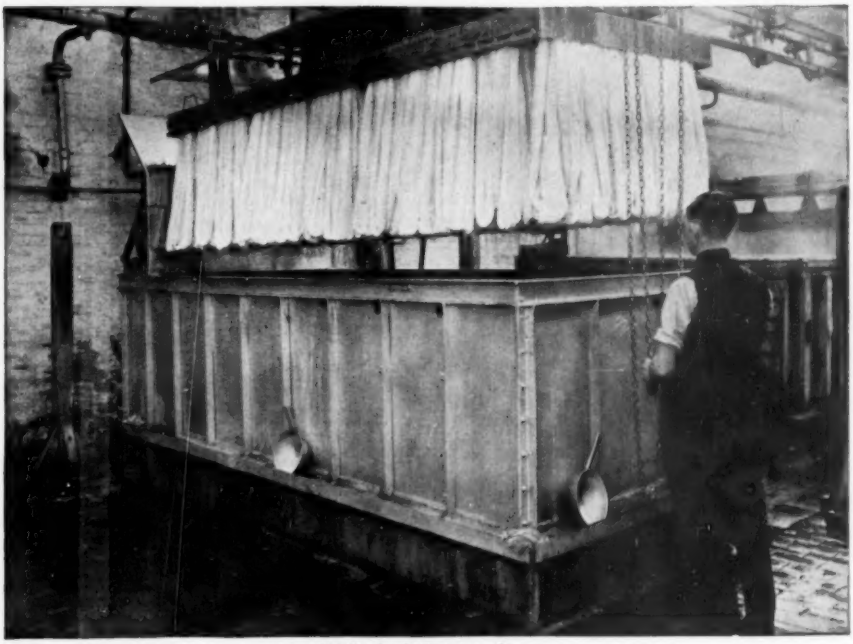

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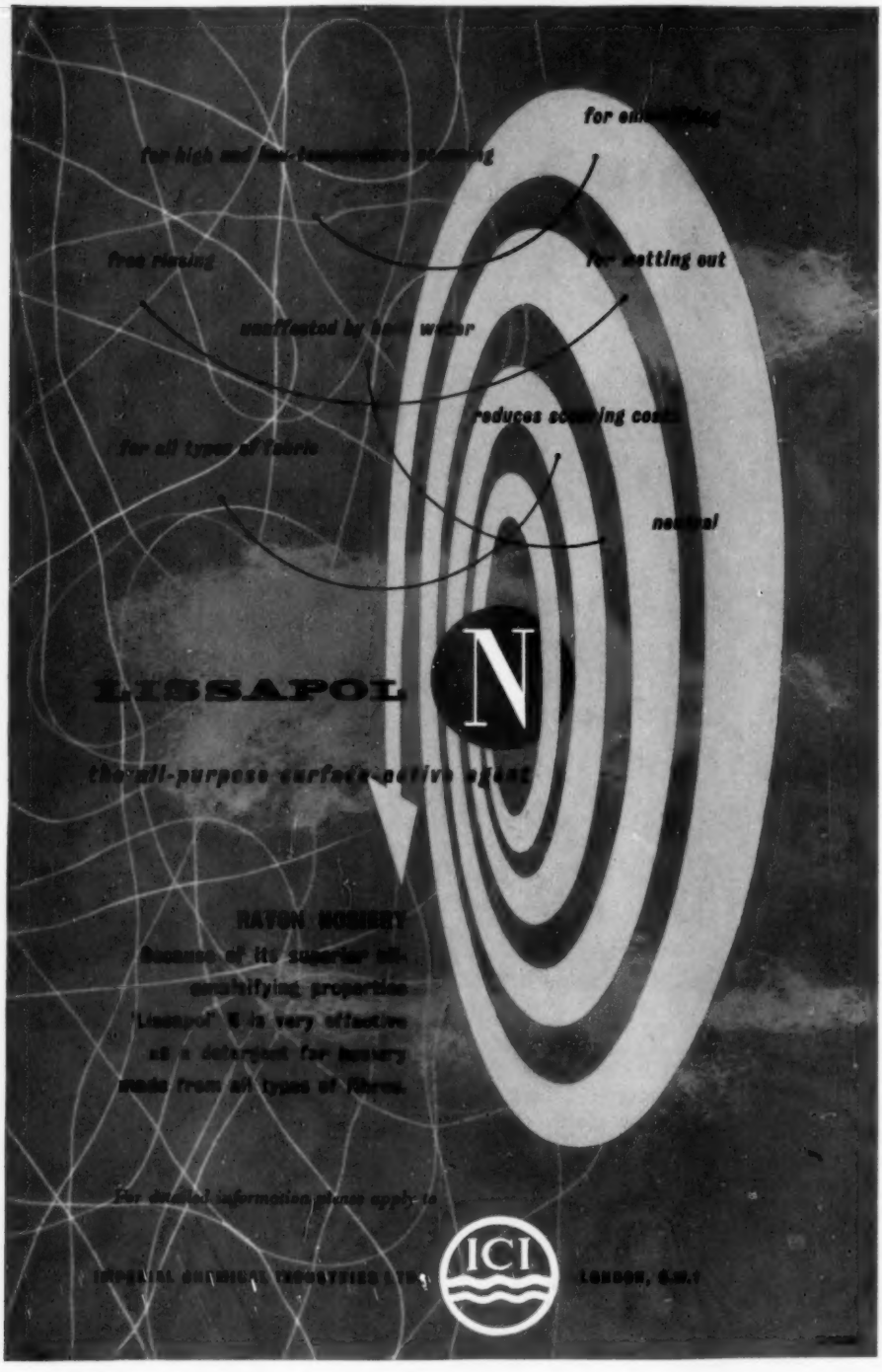
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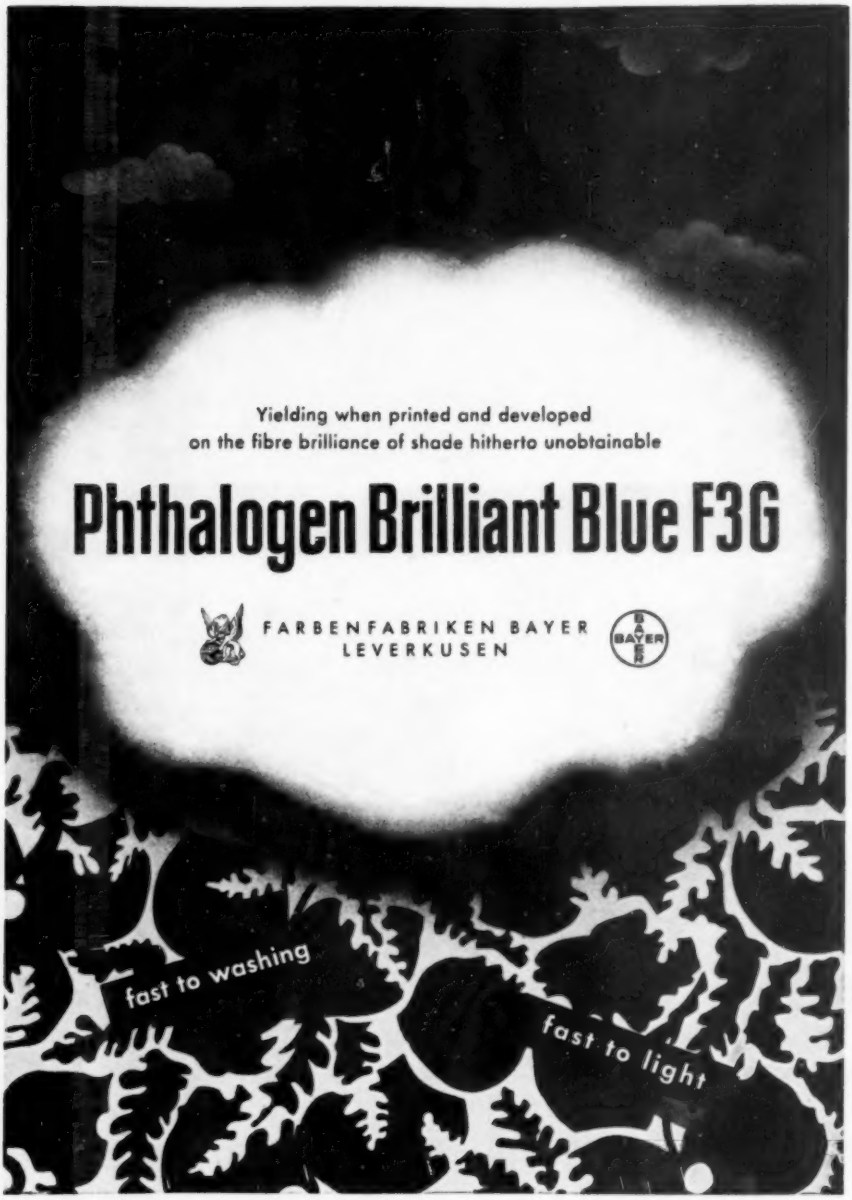
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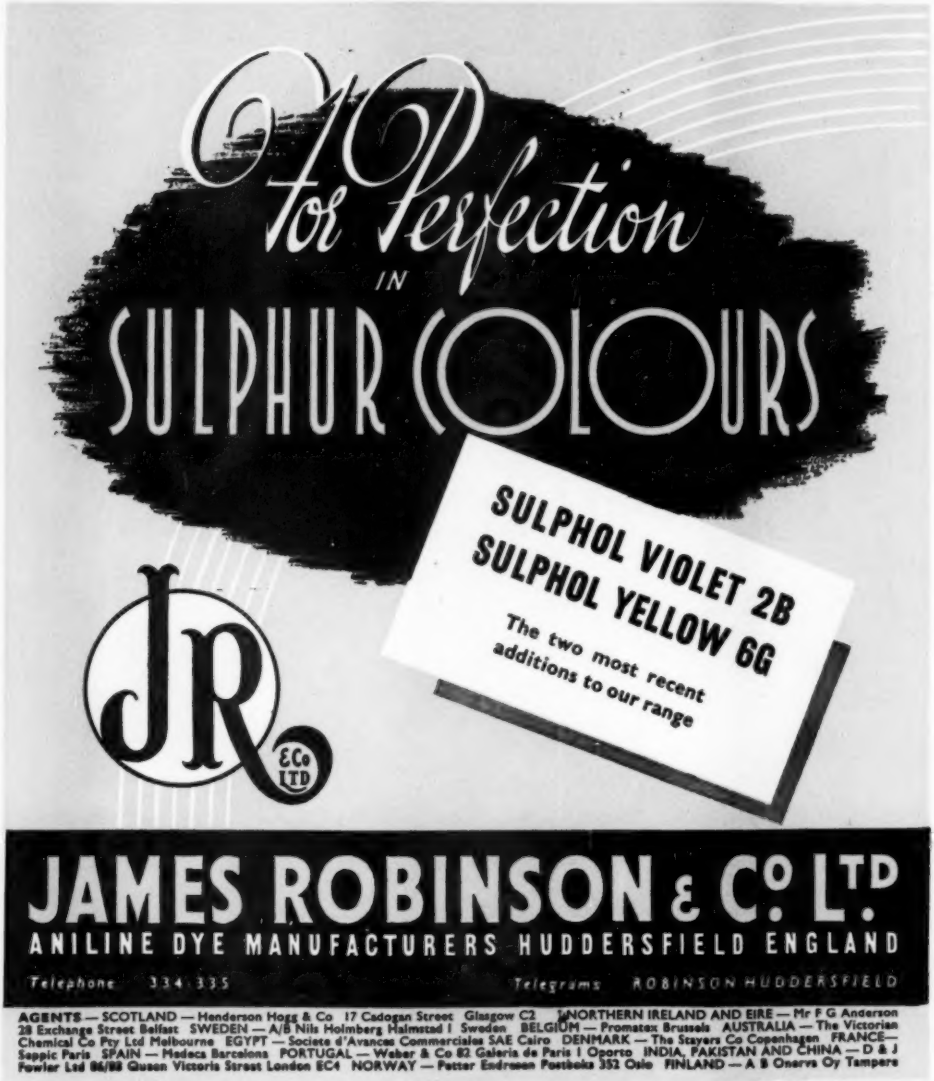

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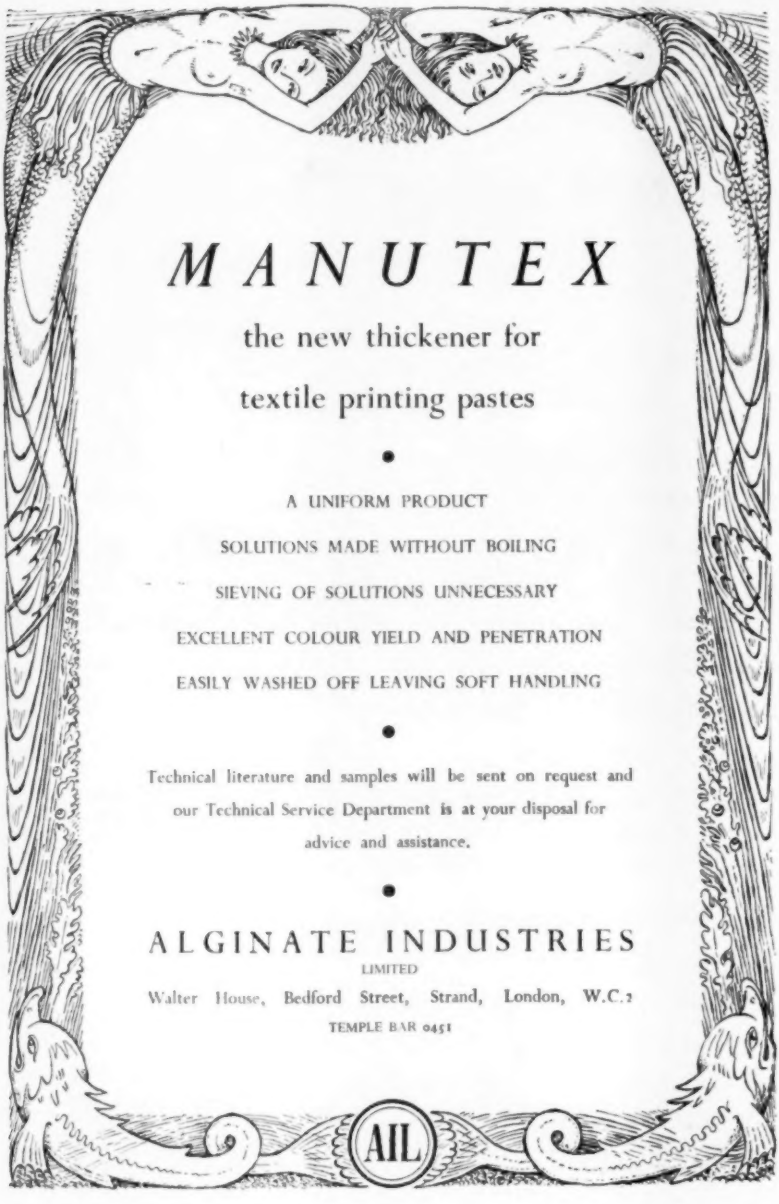

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benefit from perusing these books. The fourth volume has particular value in that it constitutes a surprising fraction of the published works devoted to the study of distillation and rectification procedures, a field in which the literature is peculiarly limited. The attention of all chemists and chemical engineers should be drawn to the new text as an exceptional compendium of theoretical considerations and valuable practice. The only regrettable thing is the price, which places the book beyond the range of most students and their preceptors.

The discussion of distillation techniques deals with all aspects of a profoundly important field of study. The theoretical consideration will probably be more useful to the chemical engineers or chemists who specialise on fractionation problems rather than to the average organic chemist. This volume has probably delved further into a specialised field than have others of the series. This should not be considered as limiting its value. The series is designed to enhance the professional and practical abilities of organic chemists, and this text will certainly achieve its object with those who are not intimidated by its aspect. The authors discuss in detail the theory and laboratory practice of fractional, extractive, and azeotropic distillation; the sublimation techniques; and the diverse equipment appropriate to distillation under wide ranges of pressure and temperature. The discussions of the laboratory apparatus include design details and recommendations for rectifying columns and their important auxiliaries which seem to cover every eventuality. One has no hesitation in deciding that this book poses the problem of whence it may be obtained rather than whether it should be consulted. J. Anderson Storrow

Abstracts from British and Foreign Journals and Patents

(The Titles of Patents are abridged and modified)

I—PLANT; MACHINERY; BUILDINGS

Factors affecting the Service Life of Leather in Wool Processing. R. M. Koppenhoefer. *Amer. Dyestuff Rep.*, 40, P 469–P 472 (23rd July 1951).

A laboratory machine and accelerated experimental procedure are described whereby the effect of wool oils on leather can be studied. Conventional leather tapes are rotated under mill speeds and tensions between grooved steel bars, wool oil being fed at intervals. Deterioration similar to that observed in practice is found, viz. hardness, transverse fissures, and splitting grains. Results may be expressed in hours to break or in loss of tensile strength after a standard running time. Spent tapes are analysed for oil content, pH, and degree of tannage. Process oil rapidly displaces natural grease from leather, and fatty-oil sulphates or mineral-oil sulphonates, which are widely used as emulsifiers, accelerate deterioration because of tanning action. Control may be effected by restricting the concentration of such components to a safe level and by avoiding excess in stock oiling. J. W. B.

PATENTS

Wet Treatment of a Linearly Moving Thread. American Enka Corpn., F. A. J. van Hall, and D. R. Barcelo. *U.S.P.* 2,539,980.

The effect of a liquid on a moving thread becomes extraordinarily intense when (1) the thread is not in contact with the machine between entering and leaving the bath, (2) the ascending thread is contacted with a stream of liquid moving in a counter-current direction, (3) the filaments forming the thread are not twisted and are parallel to one another, (4) the thread is under low tension, and (5) the thread ascends vertically. C. O. C.

Yarn-treating Vat having Guide Wheel driven by a Magnetic Coupling. American Enka Corpn. and J. K. Whisnant. *U.S.P.* 2,539,982.

Banking Reel for Rayon. American Enka Corpn. and W. J. D. van Dobbenburgh. *U.S.P.* 2,539,979.

Drying Rayon Cakes. American Enka Corpn. and F. B. Breazeale. *U.S.P.* 2,539,943.

Drying Granules, Threads, or Fibres. A.-B. Pluria. *B.P.* 655,403.

A container for the material, a separator for separating the liquid carried along by the gases, a pump for the drying steam, and a pipe for discharging steam developed during drying form a closed circuit. The drying steam is heated by the heat developed by the work of the pump. To start drying, the air in the apparatus is circulated and heated; after a short time the steam developed by drying the material becomes the main constituent of the circulating gas, and finally after discharge of gas from the system becomes the only constituent thereof. The liquid in the goods is carried away by the circulating gas, and is then removed in the separator; thus there is no need to hydro-extract the goods before placing them in this drier. The system is particularly suitable for drying packages of textiles. C. O. C.

Drying Yarn. American Enka Corpn. and P. van Dijk. *U.S.P.* 2,539,978.

Each thread is carried through a drying zone while the draw-off rate is controlled as a function of the magnitude of thread sag between supporting points placed outside the drying zone. C. O. C.

Constant-speed Automatic-reverse Tensionless Dye Jigs. P. Smith & Co. (Whitworth) Ltd., F. Leach, and I. W. Sutcliffe. *B.P.* 654,627.

In a machine of the type where each driving roller has an independently operable changeover mechanism actuated by the driving roller to reverse automatically the drive of the winding rollers when the cloth is nearly exhausted from one of them, each changeover mechanism comprises two side-by-side discs driven at differing speeds. Each disc has a notch in its periphery, the notches being engaged by a pin when in alignment. The notch in the fast running disc is V-shaped and that part of the pin engaging the notch is circular, so that when the discs start to rotate the pin will be lifted out of the notches. C. O. C.

Treating Textiles and the like in Molten Metal. Standfast Dyers & Printers Ltd., R. S. E. Hannay, and W. Kilby. *B.P.* 655,415.

When treating textiles with molten metal contained in a narrow elongated passage, the walls of the passage contain the main heating elements for the metal and are so spaced that the distance from the material being treated to the surface of any main heating element is ¼–1 in. Further heating elements may be arranged in the passage parallel to the plane of the material and at a substantial angle across its direction of motion so as to receive across their outer surfaces eddy currents of molten metal set up by the movement of the material. These elements are set so that they are ¼–1½ in. from the nearest material. This ensures uniform heat treatment of the material. C. O. C.

Drying Cylinder. Blaw-Knox Co. and H. E. Neubauer. *U.S.P.* 2,542,287.

Heated Calender Rolls. Armstrong Cork Co. *B.P.* 655,052.

A calender roll having a thin wall has two sets of heat-transfer fluid inlet ports spaced diametrically opposite each other and two similar sets of outlet ports. These sets of ports extend along the entire length of the roll. This allows the heat-transfer fluid to enter and leave from two

portions of the roll diametrically opposite one another and so enables more accurate control of the temperature across the face of the roll. C. O. C.

Printing Table. F. Buser. *B.P.* 654,713.
Two endless metal bands extend along the edges of a conveyor belt and run over feeding wheels which are mounted to rotate about the axes of the belt-supporting rollers. The bands are connected to the belt edges so as to move it along. They are guided in feeding direction between the feeding wheels by two parallel guide bars each engaging one of the bands, so that the belt is prevented from sagging and forms a taut surface on which the material to be printed is uniformly supported. C. O. C.

Web-folding Machine. Allis-Chalmers Manufacturing Co. and O. L. Strauss. *U.S.P.* 2,540,844.

Laundry Washing Machine. Prosperity Co. Inc., E. Davis, and W. F. Gayring. *U.S.P.* 2,540,431.
The mixing tank for the detergent liquor is at one end of and level with the machine, the liquor being transferred by air pressure applied to its surface. The containers for the various ingredients are readily accessible from the floor, and when the air pressure is turned on the mixing tank is automatically closed. C. O. C.

Washing Machine. O. J. Eggleton. *B.P.* 655,221.
The goods are placed on an intermittently travelling conveyor, where they are sprayed with the cleansing liquors. Pads are mounted above the conveyor. A reciprocating up-and-down movement is applied to either the pads or the conveyor, but preferably the latter, so that the goods are brought into contact with, and rubbed by, the pads. C. O. C.

Sleeve Press. Prosperity Co. Inc. *B.P.* 655,208.

Padding for Dry-cleaner Steam Presses. Patek & Co., C. G. Young, and E. C. Holler. *U.S.P.* 2,539,714, *U.S.P.* 2,539,715.

Asbestos–Nylon Cover Cloth for Garment Presses. United States Rubber Co., H. E. Sunbury, and W. C. Hitchcock. *U.S.P.* 2,542,297.

Blocking Felt Hats. H. McLachlan & Co. Inc. and H. W. Hunter. *U.S.P.* 2,542,676.

Continuous Treatment of Vegetable Fibres. Cellulose Development Corpn. Ltd. and F. G. L. Becker. (VII, p. 393.)

II—WATER AND EFFLUENTS

PATENTS

Threshold Treatment of Water. Hall Laboratories Inc. and G. B. Hatch. *U.S.P.* 2,539,305.
The small amount of metaphosphate or other molecularly dehydrated phosphate radical which need be supplied to water to inhibit precipitation of calcium carbonate on heating or addition of alkali is simply and efficiently supplied by flowing the water in contact with a bed of molecularly dehydrated phosphate which dissolves only very slowly in water. C. O. C.

Water Softening with Cation- or Anion-exchange Materials. Filtrators Ltd. and E. L. Streatfield. *B.P.* 654,559.
Apparatus is described in which samples of water are withdrawn from inside the bed of exchange material and from further on in the bed or after the water has passed out of the bed. Difference in the relative pH values of the two samples or in their relative electrical resistances indicates whether the bed is approaching exhaustion. C. O. C.

Purifying and Clarifying Water. Johns-Manville Corpn. and L. E. Weymouth. *U.S.P.* 2,542,743.
The raw water is treated by forming within it a flocculent precipitate of a hydroxide of a tervalent metal. It is then filtered through a coarse diatomaceous material. When this material becomes clogged, it is regenerated by heating it at 220-600°F. until the gel character of the metallic hydroxide is destroyed. Regenerating in this way reduces the loss of diatomaceous material to 20–10% or less of that which normally occurs. C. O. C.

III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Hydrophilic Nature of the Soda Soaps of Stearic and Palmitic Acids. A. V. Dumansky and P. A. Demchenko. *Doklady Akad. Nauk S.S.S.R.*, **78**, 277–278 (11th May 1951).
A sealed evacuated tube containing pure anhyd. sodium stearate or palmitate was broken under water or NaCl soln. in a calorimeter, and the heat of wetting was determined. The results were identical whether NaCl was present or not, and it was therefore assumed that solution effects were negligible. The degree of hydration was calculated from Dumansky's formula (according to which the percentage of combined water is $1{\cdot}25Q$, where Q is the heat of wetting in g. cal./g.). The formula is derived on the assumption that wetting proceeds through the formation of hydrogen bonds (see *Chem. Abs.*, **43**, 7781f (1949)), and is found to correspond to the monohydrate. The degree of hydration was determined also by introducing some of the anhydrous soap into a layer of xylene standing over water. The system was allowed to attain equilibrium (5–30 days), and a measured portion of the xylene layer, containing the soap, was then transferred to a Dean and Stark apparatus and the total water present determined in the usual way. After allowing for the water dissolved in the xylene, it was again found that the amount of water taken up by the soap corresponded to the formation of a monohydrate. It was concluded that the hydration and aggregation of soap molecules can be explained by the formation of hydrogen bonds. A. E. S.

Solutions of Metal Soaps in Organic Solvents. II—Peptisation and Micelle Size in Solutions of some Zinc Soaps in Toluene. V. D. Tughan and R. C. Pink. *J.C.S.*, 1804–1808 (July 1951).
Part I (*ibid.*, 1750 (1948)) reported a rapid increase in solubility of zinc soaps in non-aqueous solvents observed over a small temperature range. This critical increase was shown to be associated with a phase change in the solid soaps, and the present paper describes the effect on the critical solution temperature of a great variety of organic compounds during the peptisation of zinc soaps in toluene. The most effective peptising agents are compounds with powerful co-ordinating groups which are capable of forming complexes with the soap in solution. Although water is a moderately efficient agent, the fatty acids corresponding to the soaps are ineffective. The action of water is therefore not due to the liberation of free fatty acid by hydrolysis. Boiling-point measurements indicate that peptisation is accompanied by a marked decrease in micelle size. H. H. H.

Probable Existence of Acid Aluminium Di-soaps. E. Back and K. E. Almin. *Acta Chem. Scand.*, **4**, 1401–1407 (1950): *Chem. Abs.*, **45**, 5427 (25th June 1951).
The mechanisms by which a fatty acid can be taken up by an Al di-soap are surveyed, and the phase equilibria between an acid solution and a solid soap are deduced thermodynamically. Comparison with earlier data (*Chem. Abs.*, **42**, 8499 (1948)) yields evidence of an acid Al distearate of composition varying from $Al(OH)R_2$ to $3Al(OH)R_2.2HR$ (R = stearate ion), which is probably a solid solution of these two. C. O. C.

White Mineral Oil for Use in the Hot-oil Dyeing Process. R. K. Rhodes. *Amer. Dyestuff Rep.*, **40**, 489–490 (6th Aug. 1951).
Success of the hot-oil process depends largely on the oil adopted for the purpose, and the type of white mineral oil considered best is defined. Refinement, removal of unsaturated components, and the U.S. Pharmacopœia acid tests are described. The significance of viscosity, flash and fire points, and heat stability are discussed. It is considered that a colourless, odourless, U.S.P. acid test white mineral oil of low viscosity, high flash point, and good heat stability should be desirable for this purpose. J. W. B.

Fractionation of Starch. V—Determination of the Molecular Weights of the Triacetates of Amyloses and Amylose Fractions by Osmotic Pressure Measurements. R. S. Higginbotham. *J. Textile Inst.*, **42**, T 235–T 248 (June 1951).
The essential requirements for accurate osmometry are

discussed and an improved osmometer is described, with an account of measurements on solutions of the triacetates of samples of pure amylose and two series of fractions, from sago and tapioca amylose respectively. Nitroethane is mostly used as solvent; chloroform or 1-nitropropane to a lesser extent. Nitroethane and 1-nitropropane give the same value of $(\Pi/C)_{C \to 0}$; the values with chloroform differ, but at 25°C. the ratio of the above value in chloroform to that in nitroethane is constant over a wide range of mol. wt. and = 1·25. It is suggested, while admitting the weakness of such explanation, that this difference may be due to a small proportion of the solute being present as stable aggregates which do not dissolve on dilution, such aggregation occurring with both solvents or with nitroethane only. J. W. B.

Fractionation of Starch. VI—Fractionation of Amyloses. D. Goodison and R. S. Higginbotham. *J. Textile Inst.*, **42**, T 249–T 273 (June 1951).

A general method for fractionating amyloses is developed and is based on the discovery that their fractional solubilities in water saturated with butanol are functions of temperature and molecular size over a small temperature range. The rigidities of aqueous gels of the fractions of sago and tapioca amyloses and the intrinsic viscosities and number-average degrees of polymerisation of their triacetates are determined and compared with corresponding data for one sample of maize amylose and two samples of maize crystalline amylose. Relations between intrinsic viscosity and degree of polymerisation and between gel rigidity and degree of polymerisation differ for the different amyloses, the differences suggesting that the amyloses may be slightly branched, the amount of branching varying from one to another. J. W. B.

Alkyl Sulphosuccinates. H. Stüpel and A. von Segesser. *Textil-Rund.*, **6**, 255–268 (June 1951).

Data are given on the properties of alkyl sulphosuccinates, particularly the dioctyl, dihexyl, diamyl, and diisobutyl compounds. The properties covered are solubility, surface tension, interfacial tension against oil, wetting times according to the Draves test, foaming power, and stability to acids, alkalis, and hard water. F. A.

Improved Fatty Intermediates for Textile Finishing. L. J. Armstrong. *Amer. Dyestuff Rep.*, **40**, P 497–P 500 (6th Aug. 1951).

Attention is drawn to the fact that improved textile intermediates are largely due to the commercial development of the two basic processes of fractional crystallisation and distillation of fatty acids. Production of stearic acid free from unsaturated components and of oleic acid free from linoleic acid has led to the development of special colour- and odour-free finishes. Methods are also outlined for the production of fatty alcohols, amines, and amides which are available commercially as intermediates for the synthesis of finishing agents for softening, water repellency, and other textile applications. J. W. B.

Direct Synthesis of Methylbromosilanes. A. V. Topchiev and N. S. Nametkin. *Doklady Akad. Nauk S.S.S.R.*, **78**, 295–297 (11th May 1951).

A mixture of methylbromosilanes is formed when methyl bromide vapour is passed through a hot tube containing a mixture of metallic silicon (80%) and reduced copper (20%), the best yield being obtained at 330–340°C. The main product is dimethyldibromosilane, and this and methyltribromosilane are readily separated in the pure state. The reaction yields negligible amounts of trimethylbromosilane. A. E. S.

Direct Synthesis of Phenylbromosilanes. A. V. Topchiev, N. S. Nametkin, and N. M. Zhmykhova. *Doklady Akad. Nauk S.S.S.R.*, **78**, 497–500 (21st May 1951).

A mixture of $SiBr_4$ and phenylbromosilanes is formed when bromobenzene vapour is passed slowly through a hot tube containing a mixture of metallic silicon (80%) and reduced copper (20%), the best yield (56%) of organosilicon compounds being obtained at 410–420°C. The main product is phenyltribromosilane, which has not been previously described in the literature. It is a heavy colourless liquid, b.p. 130–131°C. at 12 mm. Diphenyldibromosilane and triphenylbromosilane may also be separated. A. E. S.

PATENTS

Sulphotricarboxylic Acid Compounds— Wetting, Dispersing, Emulsifying, and Frothing Agents. General Aniline & Film Corpn., P. Nawiasky, and G. E. Sprenger. *B.P.* 654,654.

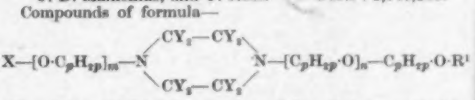
Aliphatic sulphotricarboxylic acids are prepared by heating a saturated aliphatic tricarboxylic acid with a sulphonating agent stronger than conc. sulphuric acid, or a halogen-substituted sat. or unsat. aliphatic tricarboxylic acid with an alkali-metal sulphite. Products formed have exceptional surface-active properties and are resistant to hard water. J. W. B.

Detergents and Wetting Agents. N. V. De Bataafsche Petroleum Maatschappij. *B.P.* 654,991.

Using a lime-resistant "synthetic soap" as starting material, improved wetting agents and detergents are made by forming by neutralisation or saponification in aq. synthetic soap solution a fatty acid soap, in such proportion that the fatty acid content by wt. of product is not greater than, and preferably considerably lower than, the synthetic soap content. In one example 25% aq. sodium *sec.*-alkyl sulphate solution (Teepol) (160 parts by wt.) is mixed at 60°C. with bleached palm oil fatty acids of titre 44°C. (17), 25% aq. NaOH (10) is added with stirring, followed by sodium metasilicate (45), and the mixture cooled and allowed to stand for 48 hr. A product (230) resembling soft soap is obtained. J. W. B.

***NN'*-Bispolyoxyalkylenepiperazine Derivatives having Foaming, Detergent, Wetting, and Lime-soap-dispersing Properties.** Colgate-Palmolive-Peet Co., J. D. Malkemus, and J. Ross. *U.S.P.* 2,541,260.

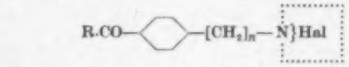
Compounds of formula—



(X = R^1 or $R^1{\cdot}CO$; R^1 = a long-chain aliphatic radical such that X contains 6–24 C; R^1 = H or short-chain Alk or acyl; the Y's = same or different H, CH_3, or C_2H_5; m and n each = 1–20; p = 2–4) are useful surface-active agents having lime-soap-dispersing properties. C. O. C.

Heterocyclic Quaternary Ammonium Compounds— Wetting Agents and Germicides. Onyx Oil & Chemical Co., A. S. Du Bois, and E. I. Valko. *U.S.P.* 2,542,642.

Compounds of formula—



(R = Alk of > 4 C; $n > 1$; the N atom is part of a one- or two-ringed heterocyclic system) are useful as cationic, germicidal, surface-active agents. C. O. C.

Strongly Foaming Detergents. General Aniline & Film Corpn., J. J. Ayo, and F. J. Gajewski. *U.S.P.* 2,542,385.

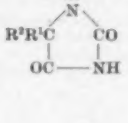
A strongly foaming liquid detergent consists of the sodium salt of oleylmethyltauride (5–25% by wt.), diethylene glycol monobutyl ether (1·25 – 12·5%), "ethylenebisiminodiacetic acid" (ethylenediaminetetraacetic acid ?) (0·5–5·0%), and water (57·5–93·25%). C. O. C.

Spinning Lubricant. Monsanto Chemicals Ltd., J. B. Storey, and M. N. Strachan. *B.P.* 654,858.

An aqueous emulsion of a colloidal solution of silica and oil, there being 7–25% by weight of oil in the emulsion, is used for lubricating cotton and rayon being spun on cotton machinery to give yarns of greater strength with less twist. C. O. C.

Hydantoin Derivatives — Textile Lubricants and Softening Agents. Publicker Industries Inc., H. Robinette, Jr., and C. R. Pacifico. *U.S.P.* 2,541,101.

The products obtained by treating a compound of formula—



(R^1 and R^2 = same or different H, phenyl, or Alk of < 6 C, $R^1 + R^2$ containing < 10 C), e.g. 5-methylhydantoin, with one of formula $R^3{\cdot}NH{\cdot}R^4$ (R^3 = aliphatic chain of 10–20 C; R^4 = H or Alk of < 10 C), e.g. dodecylamine, are textile lubricants and softening agents. They impart a full, soft, drapy handle, have excellent resistance to alkalis and washing, and do not affect the fastness properties of direct dyes. C. O. C.

Softening Agents. Ciba Ltd. *B.P.* 653,674.

Aqueous dispersions of a compound containing at least one aliphatic chain of > 11 C, e.g. olive oil, and as dispersing agent a water-soluble salt of a monoester of an aliphatic, aromatic, or *cyclo*aliphatic dicarboxylic acid with an *N*-hydroxyalkylamide, e.g. *β*-palmitoylaminoethyl hydrogen succinate, and in addition, if desired, a sulphonation product dispersing agent resistant to lime and containing an aliphatic residue of 11 C, e.g. 1-benzyl-2-heptadecylbenziminazoledisulphonic acid, are used.

B.P. 653,732.

The dispersing agent used is a water-soluble salt of the condensate of an *N*-hydroxymethylamide of a high fatty acid and a mercapto-carboxylic acid. C. O. C.

Guanidine Condensates—Softening Agents. American Cyanamid Co. *B.P.* 654,163.

Hydroxyalkyl - substituted aliphatic guanidinium aliphatic *N*-hydroxyalkylcarbamates having cationic surface-active properties and useful as softening agents are obtained by treating an aliphatic amine, having at least one H atom attached to the amino N atom, with CO_2 in aqueous or organic-solvent solution, treating the resultant amine salt with cyanamide, and condensing the resulting product with 1–12 mol. of an alkylene oxide of 2–4 C containing a single epoxy group. C. O. C.

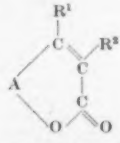
Silicon Carboxylates for Use in Coatings for Textiles, etc. Montclair Research Corpn., Ellis-Foster Co., C. A. MacKenzie, and M. Schoffman.

U.S.P. 2,537,073.

The substituted silicon carboxylates prepared from an organo-substituted silicon halide and a carboxylic acylating agent have a wide variety of uses. Thus ethylsilicon triacetate readily hydrolyses when shaken with water to produce acetic acid and a colloidally dispersed ethyl silicone. They may be used in the preparation of coatings for textiles and paper, coating compositions, etc. C. O. C.

Brightening Agents. Ciba Ltd. *B.P.* 655,258.

Compounds of formula—



(A = a benzene nucleus of which two vicinal C atoms form part of the unsat. lactone ring and which is substituted by amino (which may itself be substituted) and, if desired, other substituents; R^1 and R^2 = H, Alk, or aralkyl which may contain substituents) when sulphonated or substituted by a sulphonated radical yield brightening agents applicable to a wide variety of substrates, including all types of textiles and anodised aluminium, but they produce their best effects on animal fibres.

B.P. 655,296.

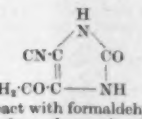
The above compounds are used in conjunction with a washing agent on fibres or films. C. O. C.

Fluorescent Resins for rendering Textiles Crease- and Shrink-resistant. Sun Chemical Corpn. and R. C. Ackerman. *U.S.P.* 2,541,184.

The products obtained by alkaline condensation of a ketone, an aldehyde, and a salt of guanidine or of a cyano derivative of guanidine when applied to cellulosic textiles and then cured not only impart shrink- and crease-resistance but also a strong fluorescence. C. O. C.

2-Keto-5-cyano-4-iminazolinecarboxyamide— Component for Resins for Treating Textiles. du Pont and D. W. Woodward. *U.S.P.* 2,535,006.

2-Keto-5-cyano-4-iminazolinecarboxyamide—



and its salts readily react with formaldehyde and hydroxymethyl compounds to form clear, colourless thermosetting resins useful for treating textiles, e.g. dimensionally stabilising rayon fabrics. C. O. C.

Polysiloxane Resins. General Electric Co. and C. D. Doyle. *U.S.P.* 2,542,641.

The resins obtained by dissolving a hydrocarbon-substituted halogenosilane in dioxan or a lower dialkyl ether of ethylene or diethylene glycol and then treating with a tertiary saturated aliphatic alcohol of 4–6 C have high viscosity and good gloss, colour, and resistance to acids and alkalis. They are useful for coating and impregnating cloth, paper, etc. C. O. C.

Alkoxychlorosilanes— Water-repellent Agents. Dow Corning Corpn. *B.P.* 653,237.

The products obtained by treating a compound of formula $R^1_{4-n}Si(OR^2)_n$ (R^1 and R^2 = Alk or Ar; n = 1–4) with a silane having bonded to the Si atom one H and at least one Cl, the remaining Si bonds not satisfied with Cl or H, if any, being satisfied by univalent Alk or Ar radicals, so as to replace at least one alkoxy radical of the first compound by a Cl atom of the silane, are water-repellent agents which contain no acidic by-products. C. O. C.

Mixed Organosiloxanes— Water-repellent Agents. Dow Corning Corpn. *B.P.* 654,099.

A mixed organosiloxane containing Si atoms directly linked to H is obtained by treating a mixture of an organohalogenosilane, having Hal and univalent hydrocarbon radicals as the only substituents attached to Si, and a mono-organodihalogenosilane, having H, Hal, and a univalent hydrocarbon radical of > 1C attached to its Si atom, with water at 0–5°c. C. O. C.

"Silicals"— Water-repellent Agents. Dow Corning Corpn. *B.P.* 654,054.

Silicals, i.e. organopolysiloxanes containing units of formula –SiHR·O– (R = Alk, aralkyl, or aromatic radical of > 4C), are obtained by hydrolysing compounds of formula $RSiHal_2H$ at not > 5°c. They are of use *inter alia* as water-repellent agents. C. O. C.

Organosilicon Waterproofing Agents. Linde Air Products Co. and L. J. Clapsadle. *B.P.* 654,450.

Compounds of formula $R^1_xSi(O{\cdot}R^3)_y(O{\cdot}R^3{\cdot}NH{\cdot}R^4)_{4-x-y}$ (R^1 and R^3 = Alk; R^3 = alkylene; R^4 = H or amino-subst. alkylene; x and y = 1 or 2, $x + y$ = 2 or 3) are obtained by refluxing together compounds of formula $R^1_xSi(OR^3)_{4-x}$ and $HO{\cdot}R^3{\cdot}NH{\cdot}R^4$ respectively. Textiles coated with a 1–10% solution of these compounds and then dried at 80°c. for 1 hr. are waterproofed. C. O. C.

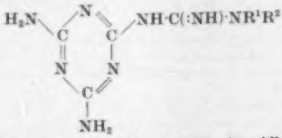
Aminoalkoxysilanes— Waterproofing Agents. Linde Air Products Co. and L. J. Clapsadle.

U.S.P. 2,541,154.

Compounds of formula $R^1_xSi(OR^3)_y(OR^2)_{4-x-y}$ (R^1 = hydrocarbon radical; R^2 = Alk; R^3 = Alk subst. by at least one amino group; x and y each = 1 or 2, $x + y$ = 2 or 3) are excellent waterproofing agents; e.g. cotton cloth is impregnated with a 3% solution in toluene of the product obtained from amyltriethoxysilane and hydroxyethylethylenediamine and heated at 80°c. for 1 hr. C. O. C.

Substituted Amidinomelamines — Flameproofing Agents and Intermediates for Dyes and Auxiliary Agents. American Cyanamid Co., D. W. Kaiser, and B. C. Redmon. *U.S.P.* 2,537,834.

Compounds of formula—



(R^1 and R^2 = same or different, H, Ar, Alk, alicyclic, aralkyl, or heterocyclic) are obtained by treating cyanomelamine with ammonia, a primary or secondary amine, or an addition salt thereof.

American Cyanamid Co. and A. F. MacLean. *U.S.P.* 2,537,840.

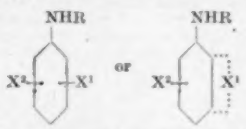
Mono-, di-, and tri-amidinomelamines are obtained by treating dicyandiamide with an anhydrous hydrogen halide. C. O. C.

Multivalent Metal Pectinate Films. U.S. Secretary of Agriculture, H. S. Owens, and T. H. Schultz. *U.S.P.* 2,542,052.

Pectinic acid of not > 7·4% methoxy content is at least partly neutralised with an inorganic hydroxide, formed into a film, and then treated with an aqueous solution of a multivalent metal salt. The films are water-resistant and have good elasticity. Those of Zn and Al pectinates are flame-resistant, while the Pb and Zn films are toxic to micro-organisms. Thus a film of Zn pectinate makes cloth flame- and mildew-resistant. C. O. C.

Biocidal Composition. General Aniline & Film Corpn. and E. M. Owen. *B.P.* 655,427.

The resins obtained by condensing formaldehyde and dicyandiamide with amines of formula—



(R = H, Alk, or Ar; X^1 and X^2 = H, Alk, Ar, Hal, halogenated alkyl, alkoxy, aryloxy, or nitro; X^1 may form a condensed ring system, e.g. quinoline or naphthalene) have bactericidal, fungicidal, and insecticidal properties. C. O. C.

Mothproofing from Dry Cleaners' Solvent. Merck & Co. Inc. and E. A. Wolff. *U.S.P.* 2,540,311.

A mixture of DDT with 2–15% of a plasticiser, e.g. dibutyl phthalate or tri-*o*-cresyl phosphate, or a mixture of a plasticiser and a resin, e.g. a methacrylate polymer, is soluble in dry cleaning solvent. Materials mothproofed with such a solution have a good handle and there is no tendency for the DDT to bloom. C. O. C.

Insect Repellents. U.S. Secretary of the Army, B. J. Magerlein, and M. S. Newman. *U.S.P.* 2,535,089.

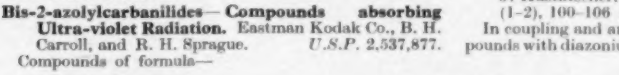
Fabric impregnated with allyl *α*-epoxy*cyclo*hexylidenepropionate remains repellent to *Aëdes aegypti* for > 10 days. C. O. C.

Insecticidal Composition. A. J.-M. Bruyneel and A. P. Chambionnat. *B.P.* 653,962.

Compounds of formula R·CH(OH)·CN (R = Alk of > 3 C) or their halogenated derivatives have good insecticidal properties and may be used *inter alia* for giving protection to furs and fabrics. C. O. C.

Bis-2-azolylcarbanilides— Compounds absorbing Ultra-violet Radiation. Eastman Kodak Co., B. H. Carroll, and R. H. Sprague. *U.S.P.* 2,537,877.

Compounds of formula—



(Z = atoms to complete a benzothiazole, thiazole, or glyoxaline nucleus; D = atoms to complete a mononuclear aryl radical; n = 1–10; m = 0 or 1) and the corresponding quaternary bisalkyl*cyclo*ammonium salts have excellent ultra-violet radiation absorption properties and may be used in coatings, filters, etc. to impart protection against ultra-violet radiation. C. O. C.

Resinous Coatings for Flexible Sheets. du Pont. *B.P.* 654,688.

Coating compositions of high resistance to abrasion and to cracking at low temp., which may be used *inter alia* in coating textile materials for heavy-duty upholstery, comprise the following ingredients— (*a*) 10–75% (by wt.) of a copolymer of butadiene and acrylonitrile, (*b*) 10–75% of a 2-halogeno-1:3-butadiene plastic polymer, and (*c*) 10–50% of a halogenated ethylene polymer. J. W. B.

Styrene–Acrylate–Maleate Copolymers. Monsanto Chemical Co. and G. R. Barrett. *B.P.* 653,443.

Copolymers of styrene or its nuclear-substituted derivatives with an alkyl acrylate, e.g. ethyl acrylate, and an alkyl half ester of an ethylene-*αβ*-dicarboxylic acid, e.g. octyl hydrogen maleate, are prepared by emulsion polymerisation. They are suitable for preparing coatings for paper, textiles, and leather. E. C.

Back-sizing Compositions for Carpets. Alexander Smith & Sons Carpet Co. and E. P. Merserau, Jr. *U.S.P.* 2,542,344.

An aqueous solution of waste sulphite liquor solids to which is added a thickening agent is used. Drying is at 200–280°F. C. O. C.

Adhesives for Bonding Rubbers or other Polymeric Materials to themselves or to Fibrous Materials. I.C.I. Ltd., T. J. Meyrick, E. G. Parry, and J. T. Watts. *B.P.* 654,331.

Compounds of formula $R^1(NH{\cdot}CO{\cdot}CR^2R^3{\cdot}CO{\cdot}R^4)_n$ (R^1 = an n-valent aliphatic, *cyclo*aliphatic, aromatic, or aliphatic-aromatic hydrocarbon radical, which can be sat. or unsat., unsubst. or subst. with a group free from active H; R^2 and R^3 = same or different, H, X^1, $CO{\cdot}X^2$, or $COOX^2$; R^4 = X^4 or $O{\cdot}X^2$; X^1, X^2, X^3, X^4, and X^5 = same or different, sat. or unsat., subst. or unsubst., univalent aliphatic, *cyclo*aliphatic, aromatic, or aliphatic-aromatic hydrocarbon radicals; n = 2–5) are used, e.g. *NN'*-bisacetoacetylhexamethylenediamine. C. O. C.

Gas-fading Inhibitors— 1:4-Bisarylmethylpiperazines. General Aniline & Film Corpn., H. W. Grimmel, and A. Guenther. (VIII, p. 394.)

Gas-fading Inhibitors. General Aniline & Film Corpn., A. F. Strobel, and W. W. Williams. (VIII, p. 394.)

Gas-fading Inhibitors— *NN'*-Bissulphomethyl-*NN'*-diarylalkylenediamines. du Pont, D. E. Kvalnes, and C. W. Croco. (VIII, p. 394.)

Polymerised Haloprene Compositions. du Pont. (XIII, p. 398.)

IV— RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

Oxidation of Toluene by Sodium Dichromate. R. D. Abell. *J.C.S.*, 1379–1381 (June 1951).

Oxidation of toluene by sodium dichromate and sulphuric acid, using a relatively small amount of oxidising agent so that the excess of toluene acts as solvent for the oxidation products, gives, besides benzoic acid and benzaldehyde, mainly phenyl *o*-, *m*-, and *p*-tolyl ketone and phenyl-*o*-, -*m*-, and -*p*-tolylmethane (these two sets of products in the ratio of 2 : 1), and very small amounts of benzyl alcohol, dibenzyl, diphenylmethane, and anthraquinone. H. H. H.

Reaction of Aromatic Diazonium Salts with Unsaturated Compounds. W. H. Brunner and J. Kustatscher. *Sitzungsber. Öster. Akad. Wiss.*, **160**, (1–2), 100–106 (1951).

In coupling and arylation reactions of unsaturated compounds with diazonium salts, viz. for styrene, *αβ*-dimethylstyrene, and *p*-methoxy-*αβ*-diethylstyrene (3-anisyl-3-hexylene) with diazotised aniline, *p*-chloroaniline, *p*-nitroaniline, and *p*-anisidine, it has been found that polarisation of the double bond is necessary for reaction, and also that the conjugation of the double bond in *αβ*-position with the phenyl nucleus is sufficient to bring it about. H. H. H.

Cleavages by means of Diazonium Compounds. IX— Phenol–Sulphur Compounds. E. Ziegler and G. Zigeuner. *Sitzungsber. Öster. Akad. Wiss.*, **160**, (3–4), 238–244 (1951).

The stabilities of certain phenol–sulphur compounds towards diazonium salts have been investigated with results as follows— 4:4'-dihydroxydiphenyl sulphide reacts with *p*-nitrobenzenediazonium sulphate in aqueous NaOH to form 2:4-bis-*p*-nitrobenzeneazophenol, whereas 4:4'-dihydroxydiphenyl sulphoxide and sulphone remain unaltered. 4:4'-Dihydroxy-3:3'-dimethyldiphenyl sulphide is split by *p*-nitrobenzenediazonium sulphate, while the corresponding sulphoxide, sulphone, and also the tris-hydroxytolylsulphonium chloride are unattacked. These results concerning the stability of the sulphur bridge have corresponding analogies with the corresponding carbon compounds; viz. dihydroxydiphenylmethane is split,

whereas dihydroxybenzophenone is not. Dihydroxydibenzyl thioethers are also readily cleaved by diazonium salts. H. H. H.

Present State of Knowledge concerning the Mechanism of Diazotisation. E. Abel. *Sitzungsber. Oster. Akad. Wiss.*, **160**, (3–4), 189–204 (1951).

Earlier and recent measurements of the kinetics of diazotisation of aniline are critically discussed, and it is concluded that no contradiction of any kind exists, and that within a wide range of concentration the velocity of diazotisation, whether of inorganic, aliphatic, or aromatic substances, is given by the expression—

$$-\frac{d\,[HNO_2]}{dt} = k\frac{[R{\cdot}\overset{+}{N}H_2]\,[HNO_2]^3}{[H^+]}$$

The catalytic influence of chlorine and bromine ions is also discussed. There are 50 references. H. H. H.

Indications of Geometrical Isomerism with 2:2′-Azopyridine. R. J. W. Le Fèvre and C. V. Worth. *J.C.S.*, 1814–1817 (July 1951).

The one known form (m.p. 87°c.) of 2:2′-azopyridine has a dipole moment which is more consistent with a *trans* than with a *cis* configuration, and its absorption spectrum resembles those of non-heterocyclic azo derivatives. Illumination by sunlight of solutions of azopyridine causes changes in their dielectric constants and spectra, which recall those observed for azobenzene, and suggest a partial development of the *cis* isomer. H. H. H.

***p*-Aminodimethylaniline. IV—Effect of Substituents on the Stability and Mode of Decomposition of the Diazonium Compounds.** J. H. Gorvin. *J.C.S.*, 1693–1697 (July 1951).

The stabilities of diazotised *p*-aminodimethylaniline and of its 2-chloro, 3-chloro, 3-nitro, 3-chloro-2-nitro, 2-chloro-3-nitro, 2-chloro-5-nitro, and 2:5-dinitro derivatives are discussed in terms of resonance in the diazonium ion. The decomposition of weakly acidic or neutral solutions of these compounds seems to proceed extensively through the unionised diazo form, since appreciable amounts of the simple deamination products are obtained. H. H. H.

***p*-Aminodimethylaniline. V—Effect of Substituents on the Azo-coupling Efficiency of the Diazonium Compounds.** J. H. Gorvin. *J.C.S.*, 1697–1700 (July 1951).

In Part I (*J.C.S.*, 613 (1941)), Ayling, Gorvin, and Hinkel showed that the low coupling power of diazotised *p*-aminodimethylaniline with amines and phenols is a consequence of electron-release by the $N(CH_3)_2$ group. It is now demonstrated that, whereas a single *meta* substituent either exerts no influence (Cl) or reduces the coupling power (NO_2), an *ortho* substituent markedly increases the efficiency of the reaction whether or not a *meta* substituent is present at the same time. Diazo compounds of this type show a general tendency to couple under the strongly acid conditions of Schoutissen. H. H. H.

Chlorination of 1-Chloro-2-naphthol. P. M. James and D. Woodcock. *J.C.S.*, 1931–1932 (July 1951).

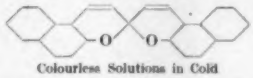
Slow chlorination of 1-chloro-2-naphthol dissolved in glacial acetic acid by the procedure of Ruggli *et al.* gave an oily product which in only three cases out of twenty-three afforded 1:4-dichloro-2-naphthol. When the acetic acid solution was kept for six days after chlorination, 1:3:4-trichloro-2-naphthol crystallised, whilst on one occasion 1:1:3:3:4-pentachloro-1:2:3:4-tetrahydro-2-ketonaphthalene was isolated; the 1:1:3:4-tetrachloro-1:2:3:4-tetrahydro-2-ketonaphthalene has also been isolated, but was more reliably prepared by chlorination of 1-chloro-2-naphthol in carbon tetrachloride. Oxidation of the various products gave phthalic acid, and there was no evidence of substitution in the 6-position, the claim of Ruggli *et al.* to have obtained 1:6-dichloro-2-naphthol not being substantiated. H. H. H.

Electron-gas Model for the Quantitative Interpretation of the Light Absorption of Organic Dyes. IIA—Evaluation of the Intensities of Absorption Bands. H. Kuhn. *Helv. Chim. Acta*, **34**, 1308–1324 (Aug. 1951).

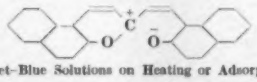
In Part I (*ibid.*, **31**, 1441 (1948)) it was shown that the positions of the absorption bands of symmetrical polymethin dyes could be approximately calculated from an electron-gas model, if the π-electrons of the polymethin chain be assumed to form a one-dimensional free electron gas which extends along the chain. In the present Part II, the intensities (*f*-values) and the directions of polarisation of the absorption bands are evaluated, and satisfactory agreement is found in a vinylene-homologous series of cyanine dyes between the calculated and empirically derived values. The best accord is obtained, however, when all possible steric forms are assumed for the dye in solution, rather than by limiting the polymethin chain to *trans*-configurations only. H. H. H.

Some Adsorption Colours and their Significance for Thermochromic and Tautomeric Effects. II—Experiments with *spiro*Pyrans and with 1:3-Diketoindane. A. Schönberg, A. Mustafa, and W. Asker. *J. Amer. Chem. Soc.*, **73**, 2876–2879 (June 1951).

Some thermochromic *spiro*pyrans are adsorbed from their colourless, cold, benzene solutions with production of colour by dried silica gel or alumina; treatment of the coloured material with methyl alcohol (elution) causes decolorisation. The colour of the adsorbates on silica gel and alumina is similar to the colour shown by the thermochromic *spiro*pyrans in hot inert solvents, e.g.

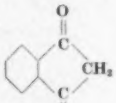


Colourless Solutions in Cold

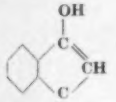


Violet–Blue Solutions on Heating or Adsorption

When alumina is introduced into the colourless benzene solution of 1:3-diketoindane (I), a violet adsorbate is formed, probably due to the enol form (II)—

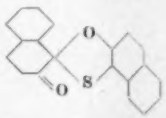


(I)

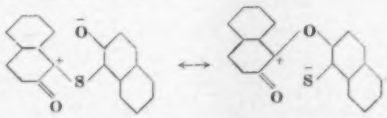


(II)

Filter paper moistened with a colourless benzene solution of 1:3-diketoindane remains colourless on drying, but on exposure to water vapour turns violet. A theory is advanced to explain these adsorption colours. Free radicals do not form coloured adsorbates on silica gel or alumina. The red dehydro-2-naphthol-1-sulphide previously regarded as the *spiro* compound—



is now regarded as a resonance hybrid—



C. O. C.

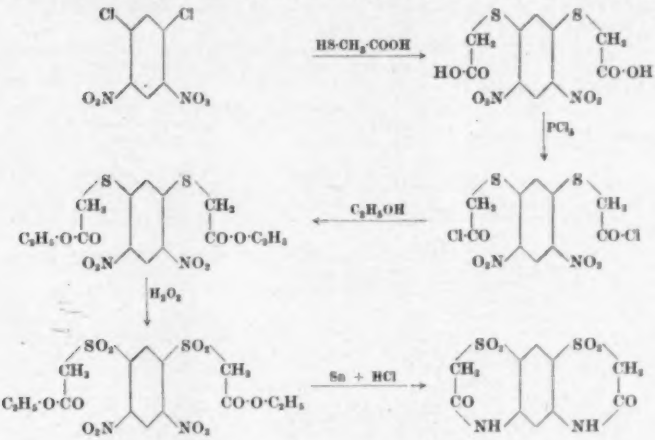
Monoazo Dyes—Absorption Spectra. J. N. Ospenson. *Acta Chem. Scand.*, **4**, 1351–1364 (1951) [in English]: *Chem. Abs.*, **45**, 4043 (10th May 1951).

The effect of *o*-CH_3, *o*- and *m*-COOH, and combinations of the two on the absorption spectra of 4-phenylazophenols and 1-phenylazo-2-naphthols was studied by coupling

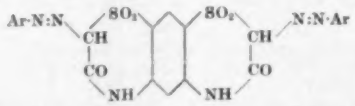
diazotised aniline, *o*-toluidine, *m*-aminobenzoic acid, anthranilic acid, 3-amino-4-toluic acid, and aminoterephthalic acid with phenol and with *β*-naphthol. The effect of the groups studied on the azo–hydrazone tautomerism and the possibility of a bifurcated hydrogen bond in the *β*-naphthol dyes are discussed. The increase in the proportion of the hydrazone form for dyes containing an *o*-carbonyl group is explained in terms of hydrogen bond formation. E. S.

Linear Disulphazone—Coupling Component for Disazo Dyes. C. Finzi and G. Leandri. *Gazzetta Chim. ital.*, **80**, 307–317 (1950): *Chem. Abs.*, **45**, 3850 (10th May 1951).

A linear disulphazone is made by the series of reactions:



In aq. NaOH suspension it yields, with 2 mol. of a diazo compound ArN_2Cl, disazo dyes—



Thus, diazotised sulphanilic acid gives a red, diazotised naphthionic acid gives a red–yellow, and diazotised Laurent acid a yellow. E. S.

Acetylenic Dyes. C. Dufraisse, A. Étienne, and P. Barbieri. *Compt. rend.*, **232**, 1043–1061 (1951): *Chem. Abs.*, **45**, 5407 (25th June 1951).

Absorption curves over the range 2000–8000 A. are given for the salts of Malachite Green, bis(*p*-dimethylaminophenyl)(phenylethynyl)carbinol, dimethylaminophenyl-(dimethylaminophenylethynyl)phenylcarbinol, Crystal Violet, bis(dimethylaminophenyl)(dimethylaminophenylethynyl)carbinol, and dimethylfuchsonimine and its ethynyl derivative (C:C not conjugated with the imine). Introduction of an acetylenic group into the active resonating system of the dye caused a large bathochromic effect on the peak in the visible range of the spectrum. Treatment of $(C_6H_5)_2C(OH)\text{-}C{:}C\text{-}C_6H_5$ with acetic acid did not give a coloured salt but an *αβ*-unsatd. ketone by addition to the triple bond and rearrangement. C. O. C.

Reactions of Diaryldicoumaranonyls and 2:2′-Diphenylthioindigo-white. A. Mustafa and A. M. Islam. *J.C.S.*, 1616–1617 (June 1951).

The action of phenylmagnesium bromide on 2:2′-diphenylthioindigo-white yields 2:3-dihydro-3-keto-2-phenylthionaphthen and diphenyl, and is an example of reductive fission of the C–C linkage by the Grignard reagent. H. H. H.

Acenaphthene Series. V—Preparation and Orientation of the Three Nuclear-substituted Mono-*tert.*-butylacenaphthenes. Isolation of Pure 1-*tert.*-Butylnaphthalene. E. Illingworth and A. T. Peters. *J.C.S.*, 1602–1607 (June 1951).

Although acenaphthene with *tert.*-butyl chloride and aluminium chloride in carbon disulphide gives 2-*tert.*-butyl- and 2:5-di-*tert.*-butyl-acenaphthene, use of anhydrous ferric chloride as catalyst unexpectedly yields 1- and 3-*tert.*-butyl- and 1:6-di-*tert.*-butyl-acenaphthene. These products are orientated by synthesis or degradation, and many new derivatives, including thioindigoid vat dyes, e.g. the red 1′-*tert.*-butyl-2:7′-thionaphthenacenaphthenylindigo, are described. 3-*tert.*-Butylacenaphthene has been converted into 4-*tert.*-butylnaphthalic anhydride and thence into pure 1-*tert.*-butylnaphthalene, which has also been synthesised from 1-tetralone by a Grignard reaction. H. H. H.

Formation and Fission of Quaternary Salts of Heterocyclic Bases containing Reactive Alkylthio Groups. D. J. Fry and J. D. Kendall. *J.C.S.*, 1716–1722 (July 1951).

The formation of quaternary salts by the action of methyl and ethyl iodides on 2-methylthio- and 2-ethylthio-benzothiazole, -benzoxazole, -pyridine, and -quinoline and the isomeric thiones has been studied in detail, and the reactions of methyl and ethyl toluene-*p*-sulphonates with these benzothiazole and quinoline compounds are also described. Contrary to published statements, the anomalous quaternisations previously observed for certain of these reactions lead, not to single compounds, but to a mixture of quaternary salts. A mechanism for these anomalies is postulated, and additional examples are included. Attention is drawn to the twofold reactivity shown by the 2-alkylthio group in the quaternary salts. H. H. H.

Action of Heat on Heterocyclic Bases containing an Alkylthio Grouping. I—2-Alkylthiobenzothiazoles and 2-Alkylthiobenzoxazoles. D. J. Fry and J. D. Kendall. *J.C.S.*, 1723–1726 (July 1951).

2-Methylthiobenzothiazole and 2:3-dihydro-3-methylbenzothiazole-2-thione on boiling undergo pyrolysis to give, amongst other products, benzothiazole and 2-methylbenzothiazole. Pyrolysis of 2-ethylthiobenzothiazole also gives benzothiazole and 2-methylbenzothiazole, but no detectable amount of 2-ethylbenzothiazole. The last can be readily identified by condensation with 2-methylthiobenzothiazole and methyl toluene-*p*-sulphonate, when not only the expected 3:3′:8-trimethylthiacyanine but also the 3:3′-dimethylthiacyanine were obtained, from which the corresponding thiacyanine dyes were prepared. 2-Methylthio- and 2-ethylthio-benzoxazoles on boiling yield the isomeric thiones but no benzoxazole or 2-methylbenzoxazole. H. H. H.

Colour of Organic Compounds. III—New Method of Assessing the ± *M* Effect of Heterocyclic Nuclei. E. B. Knott and L. A. Williams. *J.C.S.*, 1586–1589 (June 1951).

The ± *M* effects of various heterocyclic nuclei found in cyanine dyes may, with certain exceptions, be assessed in order of increasing strength by a consideration of the absorption shifts resulting on replacement of methin groups by nitrogen atoms in the chromophoric chain of dyes containing these nuclei. In general, the order is in fair agreement with that found by Brooker. The exceptions are shown by the low values of the shift for the

1-ethyl-2-quinoline nuclei, the high value for the 3-ethyl-4-methylthiazole nucleus, and the reversed order of benzothiazole and benzoselenazole. H. H. H.

Some Trinuclear Cyanine Dyes. IV—Action of Acid on Dinuclear Intermediate Dyes. F. Hamer. *J.C.S.*, xvii (June 1951).

Correction to previous paper, *ibid.*, 294–299 (Feb. 1951) (cf. J.S.D.C., **67**, 281 (July 1951)).

Red Dyes from 2:5-Dimercapto-1:3:4-thiadiazole. E. Ziegler and N. Kreisel. *Monatsh.*, **81**, 848–849 (1950): *Chem. Abs.*, **45**, 4239 (25th May 1951).

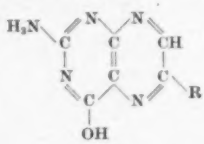
2:5-Dimercapto-1:3:4-thiadiazole (3 g.) dissolved in 10% NaOH (16 c.c.) was heated for 2 hr. at 50°c. with 37% H·CHO (3·6 g.) and acidified, when a 90% yield of 2:5-bis-(hydroxymethylmercapto)-1:3:4-thiadiazole was obtained. This was converted by the Schotten–Baumann method into the dibenzoate of the original compound. This dibenzoate (1 g.) was boiled with aniline (3 c.c.) to yield 2-mercapto-5-(*p*-aminobenzylmercapto)-1:3:4-thiadiazole, which on diazotising and coupling with phenol and β-naphthol gave a deep red dye. C. O. C.

One-electron Oxidation of Copper Phthalocyanine. A. E. Cahill and H. Taube. *J. Amer. Chem. Soc.*, **73**, 2847–2851 (June 1951).

Oxidation of tetrasulphonated copper phthalocyanine in water by certain agents produced a fugitive red intermediate with a life of several seconds. One-electron oxidising agents of sufficient potential produced this intermediate most effectively. A similar stage is seen in the oxidation of the Co (II), Fe (III), Al (III), and Zn (II) complexes but not of metal-free sulphonated phthalocyanine. In 85% H_3PO_4, the half-life of the intermediate derived from tetrasulphonated copper phthalocyanine is approx. 15 min. at 25°c. The spectrum of this red intermediate differs from that of the parent copper complex. It is shown that a net one-electron charge produces the intermediate quantitatively. The red compound disproportionates, regenerating in part the original compound, or, if excess oxidising agent is present, it undergoes net oxidation by a further one-electron charge. C. O. C.

Pteridines. III—Unambiguous Syntheses of Xanthopterin and 2-Amino-4-hydroxy-6-methylpteridine. W. R. Boon and T. Leigh. *J.C.S.*, 1497–1501 (June 1951).

Two of the key compounds in the study of pteridine derivatives of natural origin are xanthopterin (I: R = OH) and 2-amino-4-hydroxy-6-methylpteridine (I: R = CH_3), and these have now been first obtained synthetically by completely unequivocal methods.



(I)

H. H. H.

Chemistry of Adrenochrome. II—Some Analogues and Derivatives. J. D. Bu'Lock and J. Harley-Mason. *J.C.S.*, xvii (June 1951).

Correction to previous paper, *ibid.*, 712–716 (March 1951) (cf. J.S.D.C., **67**, 282 (July 1951)).

PATENTS

Stabilising Benzyl Chloride. Monsanto Chemical Co. and E. G. Somogyi. *U.S.P.* 2,542,216.

Addition of 0·05–2·0% of *cyclo*hexanol effectively stabilises benzyl chloride to decomposition due to contamination by iron. C. O. C.

Stabilised Benzyl Chloride. Monsanto Chemical Co. and J. L. West, Jr. *U.S.P.* 2,542,225.

Addition of 0·05–2·0% by weight of an *NN'*-dialkylthiourea (Alk of 1–8 C) effectively stabilises benzyl chloride to decomposition due to contamination by iron. C. O. C.

Cyanoacetamides as Azo Coupling Components. General Aniline & Film Corpn., W. H. von Glahn, and L. N. Stanley. *U.S.P.* 2,537,001.

Compounds of formula $CN\cdot CH_2\cdot CO\cdot R$ (R = NH_2, arylamino, aralkylamino, α-iminodihydro-heterocyclic (e.g. -quinolyl), ureido, thioureido, guanidino, cyanamido, and urethano) are good azo components for diazotype reproduction. Use for shading in combination with phloroglucinol or 2:3-dihydroxynaphthalene results in production of excellent neutral blacks. C. O. C.

Aliphatic Nitro Compounds as Azo Coupling Components. General Aniline & Film Corpn. and F. W. Neumann. *U.S.P.* 2,541,886.

Aliphatic nitro compounds having a nitro group attached to an aliphatic C atom carrying at least one active H atom react with light-exposed light-sensitive diazo compounds to yield a variety of hues. C. O. C.

***m*-(3-Methylsalicylamido)benzaldehyde Polyvinyl Acetals—Photographic Colour Couplers.** du Pont and E. L. Martin. *U.S.P.* 2,538,257.

3:3′:5:5′-Tetrahydroxydiphenyl as Coupling Component in Diazotype Layers on Plastic Surfaces. General Aniline & Film Corpn. and F. W. Neumann. *U.S.P.* 2,542,560.

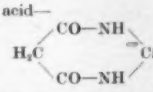
3:3′:5:5′-Tetrahydroxydiphenyl is a suitable coupling component for use in diazotype layers on cellulose ester film and other plastic surfaces. Neither it nor the dyes formed from it tend to migrate or diffuse into the plastic. C. O. C.

2:2′:4:4′-Tetrahydroxydiphenyl as Diazotype Coupling Component. General Aniline & Film Corpn., G. W. Pedlow, Jr., and F. W. Neumann. *U.S.P.* 2,542,566.

Use as a coupling component of 2:2′4:4′-tetrahydroxydiphenyl results in images of good density and excellent fastness to light and water, which do not diffuse into plastics. Coupled with *p*-phenylenediamine diazo compounds they yield deep browns. C. O. C.

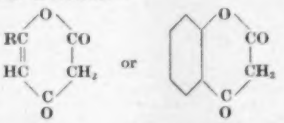
Diazotype Couplers. General Aniline & Film Corpn., W. H. von Glahn, and L. N. Stanley. *U.S.P.* 2,542,848.

Thiobarbituric acid—



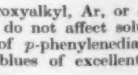
is very stable to precoupling, and with monodiazo compounds of *p*-phenylenediamines yields deep rich blues of excellent fastness to light, washing, and offset. *U.S.P.* 2,542,849.

Compounds of formula—



(R = Alk or Ar) are very stable to precoupling and do not easily oxidise to off-colour products. With *p*-phenylenediamine diazo compounds they yield bright reds to dark blues. *U.S.P.* 2,542,850.

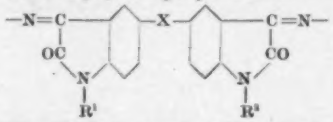
Compounds of formula—



(R = H, Alk, hydroxyalkyl, Ar, or aralkyl; X = H or substituents which do not affect solubility) with monodiazo derivatives of *p*-phenylenediamines yield bright maroons to dark blues of excellent fastness to light, washing, and offset. C. O. C.

Colour Couplers containing a Polyisatin Group Gevaert Photo-Producten N.V. and P. L. J. R. Merckx. *U.S.P.* 2,540,401.

Dyes and colour couplers are fast to diffusion in gelatin if they contain a polyisatin group of formula—



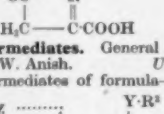
(R^1 and R^2 = H, Alk, Ar, or aralkyl; X = a bivalent atom or group, e.g. O, CH_2, $O{\cdot}CH_2{\cdot}CH_2{\cdot}O$, $CH_2{\cdot}O{\cdot}CH_2$, $CH_2{\cdot}CH_2$, in which one or more H atoms may be substituted by R^1; the benzene ring of the isatin nucleus may also contain substituents). C. O. C.

Diazotypes containing Aldehyde Reaction Products of Dihydroxyaryl Compounds. General Aniline & Film Corpn., W. H. von Glahn, and L. N. Stanley. *U.S.P.* 2,541,727.

The condensates obtained by refluxing 1 mol. of an aliphatic aldehyde with 2 mol. of a dihydroxyaryl compound in a bath made slightly acid with formic acid at < 100°c. for 2–4 hr. when used as azo dye coupling components yield sepias of excellent fastness to light. C. O. C.

Heterocyclic Couplers for Diazotype Material. General Aniline & Film Corpn. and S. C. Slifkin. *B.P.* 655,007.

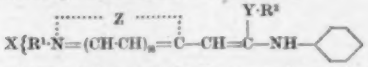
Deep reds of good fastness to light and excellent wash fastness properties are obtained by coupling diazo derivatives of *p*-diamines of the benzene series with compounds of formula—



C. O. C.

Cyanine Dye Intermediates. General Aniline & Film Corpn. and A. W. Anish. *U.S.P.* 2,536,973.

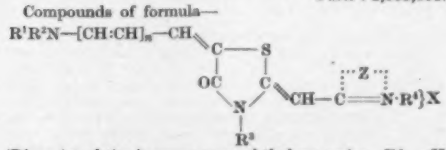
Cyanine dye intermediates of formula—



(R^1 and R^2 = Alk or aralkyl; X = an acid radical); Y = S or Se; Z = residue of a nitrogenous heterocyclic base; n = 0 or 1) are obtained by (1) treating a quaternary salt of a nitrogenous heterocyclic base (containing a reactive CH_3 group *a* to the heterocyclic quaternary N atom) with phenyl *isothio-* or *isoseleno*-cyanate in presence of an organic base and alcohol and (2) alkylating the resulting compound with an alkyl or aralkyl ester. C. O. C.

Thiazolone Cyanine Dye Intermediates. General Aniline & Film Corpn. and T. R. Thompson. *U.S.P.* 2,535,992.

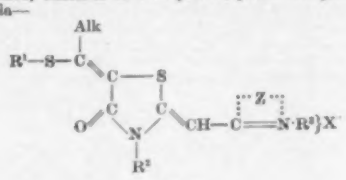
Compounds of formula—



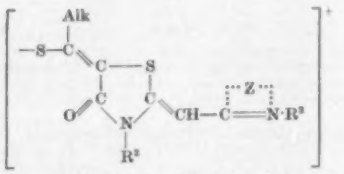
(R^1 = Ar of the benzene or naphthalene series; R^2 = H or acetyl; or R^1 and R^2 may together complete a piperidine, *β-* or *γ*-pipecoline, pyrrolidine, morpholine, or tetrahydroquinoline nucleus; R^3 = Alk, allyl, Ar, or aralkyl; R^4 = Alk, Ar, or aralkyl; n = 0, 1, or 2; X = an acid radical; Z = atoms to complete a nitrogenous heterocyclic ring) are useful intermediates in preparing polynuclear cyanine dyes. C. O. C.

Thioethers and Thioketones of Thiazolone Cyanine Dyes as Intermediates for Chain-substituted Tri- and Tetra-nuclear Cyanine Dyes. General Aniline & Film Corpn. and T. R. Thompson. *U.S.P.* 2,536,987.

Treatment of the dyes described in *U.S.P.* 2,536,986 (p. 390) with an aliphatic acid anhydride in presence of a mixture of pyridine and triethylamine followed by treatment with phosphorus pentasulphide to yield the thio derivative, which is then alkylated, yields compounds of formula—



or



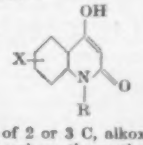
(R^1 = Alk or aralkyl; R^2 = Alk, alkenyl, Ar, or aralkyl; R^3 = subst. or unsubst. Alk, Ar, or aralkyl; X = an acid radical; Z = atoms necessary to complete a nitrogenous heterocyclic ring. Treating these compounds with heterocyclic ketomethylene compounds yields chain-substituted trinuclear cyanine dyes, while with thiazolone cyanine dyes they yield both symmetrical and unsymmetrical chain-substituted tri- and tetra-nuclear cyanine dyes. C. O. C.

Monoazo Dyes applicable by a Single-bath Process in presence of Nickel or Cobalt Salts. Ciba Ltd. *B.P.* 653,097.

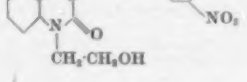
Monoazo dyes applicable to animal fibres by a single-bath process in presence of a Co or Ni compound are made by *OO'*-diacylating dyes of the type *o*-aminophenol→*β*-naphthol, in which the *o*-aminophenol contains a nitro group, or, if free from nitro groups, contains > 1 Hal, and the acylating agent contains at least one salt-forming substituent. The preferred acylating agent is the *m*-sulphonyl chloride of benzoic acid, which does not yield the expected sulphonic ester, but gives the carboxylic ester. Thus the monoazo compound 5-nitro-2-aminophenol→*β*-naphthol is esterified with the *m*-sulphonyl chloride of benzoic acid to yield a dye giving navy blue on wool when applied in presence of Glauber's salt, acetic acid, and cobalt acetate. E. S.

Yellow Azo Dyes for Cellulose Acetate, etc. derived from *N*-(subst. Alkyl)-4-hydroxy-2-quinolones. Eastman Kodak Co. and J. B. Dickey. *U.S.P.* 2,529,924.

Yellow acetate rayon dyes of low volatility are made by coupling suitable diazo compounds with an *N*-substituted 4-hydroxy-2-quinolone—



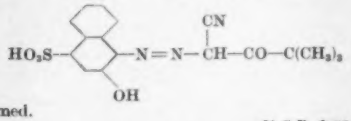
(R = hydroxyalkyl of 2 or 3 C, alkoxyalkyl of 3 or 4 C, *β*-carbamylethyl, *β*-carbomethoxyethyl, or *β*-carbethoxyethyl; X = H, Cl, Br, CH_3, OCH_3, CF_3, or NO_2). Thus *N*-*β*-hydroxyethylanthranilic acid is refluxed with acetic anhydride to yield *N*-*β*-hydroxyethyl-4-hydroxy-2-quinolone, which couples under alkaline conditions with diazotised *m*-nitroaniline to yield the yellow azo dye—



E. S.

Metallisable Azo Dyes from Trimethylacetoacetonitrile. American Cyanamid Co. and R. S. Long. *U.S.P.* 2,525,611.

Trimethylacetoacetonitrile, made e.g. by heating a mixture of ethyl trimethylacetate, acetonitrile, and sodium methoxide at 102–105°C., couples with diazo compounds having an OH or COOH group *ortho* to the diazo group to yield metallisable dyes. Thus with diazotised 1-amino-2-naphthol-4-sulphonic acid the bluish-red wool dye—

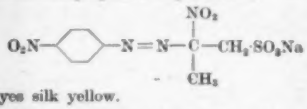


is formed. *U.S.P.* 2,525,610.

The dyes of *U.S.P.* 2,525,611 may be metallised in substance, especially with salts of chromium. Thus the dye 1-amino-2-naphthol-4-sulphonic acid→trimethylacetoacetonitrile is refluxed with basic chromium acetate in presence of H_2SO_4 to yield a bright bluish-red wool dye. E. S.

Azo Dyes from β-Nitroalkanesulphonic Acids. Visking Corpn., M. H. Gold, and H. H. Levine. *U.S.P.* 2,526,218.

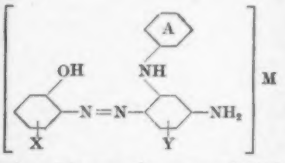
Water-soluble sulphonates of β-nitroalkanes in which the nitro group is directly attached to the carbon atom of a methylene (CH_2) or methenyl (CH) group (see *U.S.P.* 2,510,281 for preparation thereof) couple with diazo compounds at the thereby activated $-CH_2{\cdot}NO_2$ or $>CH{\cdot}NO_2$ to yield azo dyes. Thus ammonium 2-nitropropane-1-sulphonate couples with diazotised *p*-nitroaniline in presence of sodium acetate to yield sodium 2-nitro-2-*p*-nitrobenzeneazopropane-1-sulphonate—



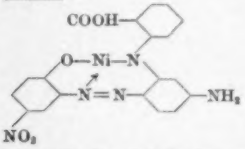
which dyes silk yellow. E. S.

Metallised Monoazo Dyes for Nylon. General Aniline & Film Corpn., H. H. Bestehorn, and J. F. Morgan. *U.S.P.* 2,529,444.

Deep dyeings of good fastness on nylon are given by dyes of formula—



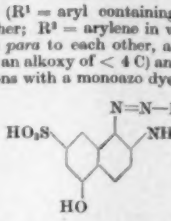
(the nucleus A contains an *ortho* or a *para* COOH; X = H, Alk, alkoxy, NO_2, or Hal; Y = H, Alk, alkoxy, or Hal; M = Cr, Cu, Co, or Ni). They are not as soluble in water as metallised dyes having a sulpho group, but their solubility is such that dispersion is not necessary for their application, and they may be dyed in admixture with more soluble metallised dyes. Thus diazotised *p*-nitro-*o*-aminophenol is coupled with *N*-*m*-aminophenylanthranilic acid in presence of acetic acid. The monoazo compound so formed is heated at 50–60°c. with nickel sulphate in presence of aq. ammonia to yield a brown dye for nylon of probable formula—



E. S.

Substantive, Metallisable, Trisazo Dyes. Ciba Ltd. *B.P.* 654,744.

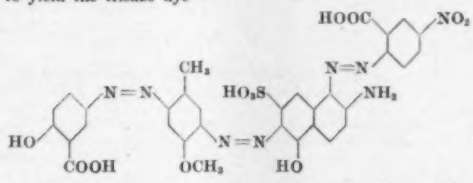
Mainly navy blue substantive dyes are made by diazotising an aminoazo dye free from sulpho groups $R^1{\cdot}N{:}N{\cdot}R^2{\cdot}NH_2$ (R^1 = aryl containing OH and COOH *ortho* to each other; R^2 = arylene in which the NH_2 and the azo link are *para* to each other, and which contains *ortho* to the NH_2 an alkoxy of < 4 C) and coupling it under alkaline conditions with a monoazo dye—



(R^3 = aryl free from sulpho groups, containing COOH or carboxyalkoxy of 2 or 3 C *ortho* to the azo link, and also a nitro group). Thus 5-aminosalicylic acid is diazotised and coupled under acid conditions with cresidine. The aminoazo dye so formed is diazotised and coupled under alkaline conditions with the monoazo compound—

5-Nitroanthranilic acid $\xrightarrow{\text{Acid}}$ 2-Amino-5-naphthol-7-sulphonic acid

to yield the trisazo dye—



It dyes cotton navy blue, rendered fast to washing and light by aftercoppering. E. S.

Non-dusting Stabilised Diazo Salts. General Aniline & Film Corpn. and H. A. Bergstrom. *U.S.P.* 2,527,347.

The dust associated with stabilised diazo salts and other dyes in powder form is reduced by admixing with 1–3% of an alkyl (< 5 C) ester of monochloroacetic acid. R. K. F.

Non-dusting Dye Powders. General Aniline & Film Corpn. and A. E. Hermann, Jr. *B.P.* 652,915.

1–3% of a liquid lower-alkyl silicone, e.g. dimethyl silicone, having a viscosity of 1–1000 centistokes at 25°c. is ground with a dye powder to give a dustless free-flowing product. R. K. F.

Super-sulphonated Anthraquinone Acid Dyes. Sandoz Ltd. *B.P.* 652,448.

Acid dyes of the types (*a*) 1:4-diamino(or subst. amino)-anthraquinone-2-sulphonic acid and (*b*) 4-arylamino-anthraquinone-1:9-pyridone-2-sulphonic acid are further sulphonated to give dyes which dye wool and nylon, only in presence of sulphuric acid. Thus 1-amino-4-(1′:2′:3′:4′-tetrahydro-2′-naphthylamino)-anthraquinone-2-sulphonic acid is treated with five times its weight of monohydrate for 1 hr. at 0°c. R. K. F.

Hydroxyalkylaminoanthraquinones—Dyes for Cellulose Esters and Ethers. Polymerisable Products Ltd. *B.P.* 655,335.

Anthraquinone derivatives or their leuco compounds, containing a replaceable group in the 1-position, are condensed with a tertiary hydroxyalkylamine of formula $HO{\cdot}CR^1R^2{\cdot}CHR^3{\cdot}NH_2$ (R^1 and R^2 = Alk or Ar, or are combined in a *cyclo*aliphatic ring; R^3 = H or Alk of < 5 C) to give dispersible dyes for cellulose acetate. Thus leuco-quinizarin is refluxed in ethanol with 3-amino-2-methyl-2-butanol for 4 hr., after which monomethylamine is added and refluxing continued for a further 6 hr. The resulting mixture is then oxidised by passing air through after adding piperidine and copper acetate. R. K. F.

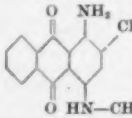
1:4-Diacylamino-6(or 7)-halogenoanthraquinones—Vat Dyes. Ciba Ltd. *B.P.* 652,452.

Pink vat dyes are obtained by acylating a 1:4-diamino-6(or 7)-halogenoanthraquinone so that one acylamino group is alkylsulphonylbenzamido and the other is free from the sulphonyl group. Thus *m*-methylsulphonylbenzoic acid is heated for 1·5 hr. at 110–120°c. with

thionyl chloride in nitrobenzene containing pyridine. 1-Benzoylamino-4-amino-6-chloroanthraquinone is then added, and heating continued at 120–130°c. for 2 hr.
R. K. F.

1 - Amino - 2 - trifluoromethyl - 4 - polyfluoroalkyl - aminoanthraquinones — Dyes for Cellulose Esters and Ethers. Eastman Kodak Co., J. B. Dickey, and E. B. Towne. *U.S.P.* 2,537,976.

Violet to blue dyes for cellulose acetate are obtained by condensing a 1-amino(or subst. amino)-2-trifluoromethyl-4-bromoanthraquinone with a polyfluoroalkylamine. Thus, 1 - amino - 2 - trifluoromethyl - 4 - bromoanthraquinone is refluxed in butyl alcohol containing copper and potassium acetates with 1:1-difluoro-2-aminopropane (prepared by hydrogenating the oxime of difluoroacetone) to give the dye—



R. K. F.

Anthraquinone Dyes for Cellulose Esters and Superpolyamides. Ciba Ltd. *B.P.* 652,453.

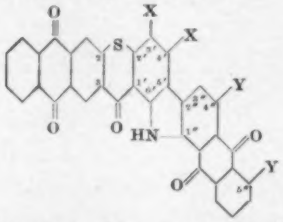
Orange to blue water-soluble dyes for cellulose acetate and nylon are made by treating a 1-amino(or subst. amino)-2-hydroxyalkoxyanthraquinone with a dicarboxylic anhydride. Thus 1 - amino - 4 - hydroxy - 2 - β - hydroxyethoxyanthraquinone is heated with maleic anhydride in pyridine for 30 min. at 60°c. The resulting red dye is separated by pouring into water, and converted to its sodium salt by drying with borax and sodium hydroxide.
R. K. F.

Iodoanthanthrones—Vat Dyes. General Aniline & Film Corpn. and D. I. Randall. *B.P.* 652,328.

Anthanthrone is iodinated or bromoiodinated by heating under pressure in oleum with iodine or a mixture of iodine and bromine. Thus anthanthrone is heated at 130°c. and approx. 75 lb./sq. in. with iodine in 3·6% oleum. The products, separated by pouring into water and purified by alkali extraction, are scarlet to violet vat dyes. R. K. F.

Anthrimide–Carbazole–Thioxanthones — Vat Dyes. General Aniline & Film Corpn., F. Max, and D. I. Randall. *B.P.* 652,386.

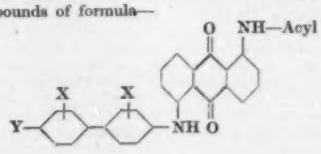
Orange to olive vat dyes of formula—



(X = H, Hal, Alk, Ar, alkoxy, or aroxy; Y = H, alkoxy, or benzamido) are prepared by condensing an anthraquinone-3:2(*S*):1′:2′(*S*)-6′-halogenothioxanthone with a 1-aminoanthraquinone and cyclising the resulting compound. Thus anthraquinone-3:2(*S*):1′:2′(*S*)-3′:6′-dichlorothioxanthone is heated with 1-amino-5-benzamidoanthraquinone at 205–210°c. for 5 hr. in nitrobenzene in presence of copper acetate and sodium carbonate. The resulting anthrimide is cyclised by heating at 74–79°c. for 1 hr. in sulphuric acid.
R. K. F.

Anthraquinone–Carbazole Derivatives—Acid Dyes. Sandoz Ltd. *B.P.* 650,928.

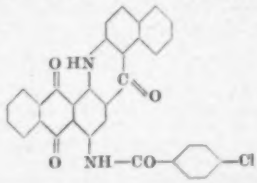
Compounds of formula—



(X = H, Alk, alkoxy, or Hal; Y = H, Alk, Hal, or acylamino) are cyclised and sulphonated to give brown dyes for wool and nylon by treating with sulphuric or chlorosulphonic acid. Thus, 1-benzoylamino-5-*p*-phenylanilinoanthraquinone is stirred with monohydrate for 3 hr. at 10–25°c., 28% oleum next added, and stirring continued until a test shows complete solubility in water.
R. K. F.

4-Acylaminoanthraquinone-naphthacridones — Vat Dyes. Ciba Ltd. *B.P.* 652,326.

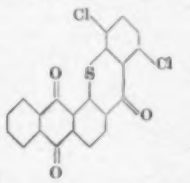
A 4-aminoanthraquinone-2:1(*N*):1′:2′(*N*)-naphthacridone (prepared by condensing a 1-halogeno-4-nitroanthraquinone-2-carboxylic acid and a 2-aminonaphthalene, cyclising, and reducing the NO_2 group) free from other NH_2 or NO_2 groups is treated with an acylating agent. Thus 4-aminoanthraquinone-2:1(*N*):1′:2′(*N*)-naphthacridone is heated with *p*-chlorobenzoyl chloride in nitrobenzene at 140–150°c. for 1·5 hr. to produce the grey vat dye—



R. K. F.

Preparation of Thioxanthones using Phosphorus Pentoxide in Phosphoric Acid. General Aniline & Film Corpn. and W. Schmidt-Nickels. *U.S.P.* 2,521,676.

Anthraquinonyl phenyl thioethers containing a carboxyl group are cyclised to the corresponding thioxanthones by heating with a solution of phosphorus pentoxide in phosphoric acid. Thus the compound—



is prepared by heating the condensation product of 1-nitroanthraquinone-2-carboxylic acid and 2:5-dichlorobenzenethiol with a solution consisting of equimolecular proportions of P_2O_5 and H_3PO_4, at 180°c. for 20 min. R. K. F.

***iso*Thiouronium Dyes.** I.C.I. Ltd., Z. E. Jolles, J. Wardleworth, and C. Wood. *B.P.* 650,850.

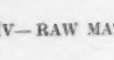
Insoluble coloured compounds of the azo, anthraquinone, phthalocyanine, and dioxazine series containing a Hal·CH_2– group linked to the aromatic residue via an –NR·SO_2–, –CO·NR–, –CO·O·CH_2–, –CH_2·NR–, or –CH_2·O·CH_2·CH_2·O– group (R = H or hydrocarbon) are converted to soluble dyes by treating with thiourea or a substituted thiourea. The resulting compounds differ from those described in *B.P.* 587,636 and 613,980 in that the *iso*thiouronium group is connected to the aromatic nucleus via one of the above linking groups instead of directly by a methylene group. Thus, the pigment obtained by coupling diazotised *p*-amino-*N*-*cyclo*hexyl-*o*-chloroacetanilide with 2-hydroxy-3-naphthoic *o*-methyl-*p*-chloroanilide is stirred at 95–100°c. for 30 min. with tetramethylthiourea and water. The resulting red water-soluble dye is precipitated by pouring into acetone.
R. K. F.

Dialkylaminobenzylidene Dyes. Eastman Kodak Co., L. G. S. Brooker, and G. H. Keyes. *U.S.P.* 2,538,008.

Dyes of formula—

HO·CO·Q·CO·NH·D^1·CO·C(CN):CH·D^2·NR^1R^2

(D^1 and D^2 = *p*-arylene groups of the benzene series; Q = a bivalent hydrocarbon radical of 2–3 C or an *o*-arylene group of the benzene series; R^1 and R^2 = same or different Alk) are obtained by treating a compound of formula H_2N·D^1·CO·C(CN):CH·D^2·NR^1R^2 with one of formula—



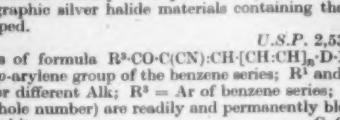
in presence of a heterocyclic base of the pyridine series. They bleach readily and permanently when exposed photographic silver halide materials containing them are developed.

U.S.P. 2,538,009.

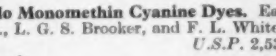
Dyes of formula $R^3{\cdot}CO{\cdot}C(CN){:}CH{\cdot}[CH{:}CH]_n{\cdot}D{\cdot}NR^1R^2$ (D = *p*-arylene group of the benzene series; R^1 and R^2 = same or different Alk; R^3 = Ar of benzene series; n = 0 or a whole number) are readily and permanently bleached by sulphites. C. O. C.

***N*-Arylthiazolo Monomethin Cyanine Dyes.** Eastman Kodak Co., L. G. S. Brooker, and F. L. White. *U.S.P.* 2,537,876.

Dyes of formula—



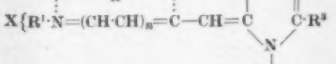
(R^1 = subst. or unsubst. Alk; R^2 = H, or subst. or unsubst. Alk or Ar; R^3 = subst. or unsubst. Alk or Ar; X = an anion; n = 0 or 1; Z = atoms to complete a heterocyclic nucleus) are obtained by condensing a compound of formula $Hal{\cdot}CHR^3{\cdot}CO{\cdot}R^3$ with one of formula—



C. O. C.

Cyanine Dyes having a Sulphohydrocarbon Radical on an Auxochromic Nitrogen Atom. Kodak Ltd. and R. H. Sprague. *B.P.* 654,690.

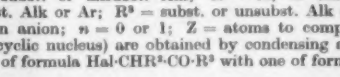
Dyes of formula—



(d and n each = 0 or 1; m = 0, 1, or 2; L = subst. or unsubst. methin; R = subst. or unsubst. Alk; D = a bivalent hydrocarbon radical; Z^1 and Z^2 each = atoms to complete a 5- or 6-membered heterocyclic nucleus) having a sulphoalkyl group on but one of the auxochromic N atoms are zwitterions, whereas similar dyes having a carboxyalkyl instead of the sulphoalkyl group contain a separate cation. Dyes of the above formula containing either one or two sulphoalkyl groups attached to auxochromic N unlike the corresponding biscarboxyalkyl dyes are insoluble in ethyl alcohol containing triethylamine. C. O. C.

Acid *mero*Cyanine Dyes. Kodak Ltd. and R. H. Sprague. *B.P.* 654,683.

Dyes of formula—



(R^1 = carboxyalkyl or sulphoalkyl; R^2 = Alk or Ar; L = methin; n = 0 or 1; m = 0–3; d = 0 or 1; Q^2 = O, S, N·Alk, or N·Ar; Z and Q^1 = atoms to complete a 5- or 6-membered heterocyclic nucleus) are mostly photographic sensitisers which are removed upon alkaline development. Those that are not sensitisers are of use as light-screening substances. C. O. C.

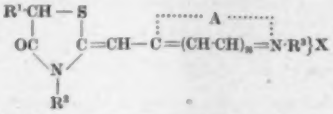
Polymethin Dyes. Eastman Kodak Co., S. G. Dent, Jr., and L. G. S. Brooker. *U.S.P.* 2,537,880.

Polymethin dyes are prepared by condensing a dialkoxymethyl carboxylate with a compound of formula—

(R = subst. or unsubst. Alk; X = an anion; n = 0 or 1; Z = atoms to complete a thiazole, benzothiazole, naphthothiazole, thionaphtheno-7′:6′:4:5-thiazole, oxazole, benzoxazole, naphthoxazole, selenazole, benzoselenazole, naphthoselenazole, thiazoline, 2- or 4-quinoline, 1- or 3-*iso*quinoline, 3:3-dialkylindolenine, 2- or a 4-pyridine nucleus; when Z = a thiazole, thiazoline, benzothiazole, or 3:3-dialkylindolenine nucleus, the R may = Ar). C. O. C.

Thiazolone Cyanine Dyes. General Aniline & Film Corpn., L. C. Hensley, and T. R. Thompson. *U.S.P.* 2,536,986.

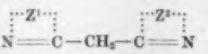
Dyes of formula—



(R^1 = H, Hal, or CH_3; R^2 = Alk, allyl, Ar, or aralkyl; R^3 = subst. or unsubst. Alk or aralkyl; A = residue of a heterocyclic nitrogenous nucleus; n = 0 or 1; X = an acid radical) are obtained by condensing a substituted thioamide with an *α*-halogeno- or *αα*-dihalogeno-acetic acid or an *α*-halogenopropionic acid. C. O. C.

***NN′*-Alkylenecyanine Dyes.** Eastman Kodak Co., L. G. S. Brooker, and R. H. Sprague. *U.S.P.* 2,541,400.

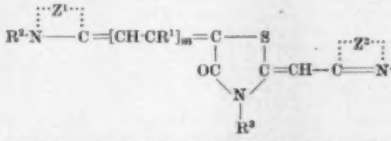
NN′-Alkylenecyanine dyes are prepared by treating an alkylene "salt" of formula $X^1{\cdot}[CH_2]_n{\cdot}X^2$ (n = 2 or 3; X^1 and X^2 each = an arylsulphonate radical) with a compound of formula—



(Z^1 and Z^2 each = atoms needed to complete a 5- or 6-membered heterocyclic nucleus) and treating the hydro salt so obtained with an acid-binding agent. C. O. C.

Trinuclear Cyanine Dyes. General Aniline & Film Corpn. and T. R. Thompson. *U.S.P.* 2,535,993.

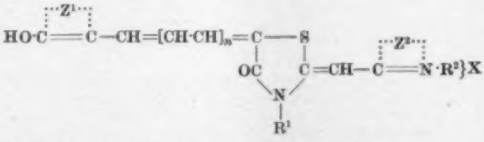
Dyes of formula—



(R^3 = H or Alk, being only Alk when m = 1; R^2 and R^4 = same or different, subst. or unsubst., Alk, Ar, or aralkyl; R^3 = Alk, allyl, Ar, or aralkyl; m = 1–3; X = an acid radical; Z^1 and Z^2 = same or different heterocyclic residues) are obtained in excellent yield and readily purifiable form by treating a *cyclo*ammonium quaternary salt, containing a reactive methyl group *α* to its N atom, with a thiazolone cyanine dye salt intermediate in presence of a basic condensing agent.

U.S.P. 2,535,994.

Dyes of formula—



(Z^1 and Z^2 = residues of heterocyclic nuclei; R^1 = Alk, allyl, Ar, or aralkyl; R^2 = subst. or unsubst., Alk, Ar, or aralkyl; n = 0–4; X = an acid radical) are obtained in excellent yield and readily purifiable form by condensing a 5- or 6-membered keto-heterocyclic compound containing a reactive mono- or poly-methin group with a thiazolone cyanine dye salt by heating in presence of a tertiary base on a steam-bath or refluxing for 5–20 min. C. O. C.

Desensitising Dyes. Eastman Kodak Co., W. B. Kendall, and G. D. Hill. *U.S.P.* 2,541,472.

Benzothiazole, quinoline, indolenine, benzotriazole, and rhodamine compounds having one or more nitro groups attached to a benzene nucleus which is either part of a heterocyclic compound or is attached to it through a doubly bonded carbon–carbon chain are desensitising dyes for use in direct positive photographic emulsions. C. O. C.

Furnace Blacks. Columbian Carbon Co., W. C. Ekholm, and G. L. Heller. *B.P.* 654,864, *B.P.* 654,865.

Packaging of Lamp Black, etc. Charles H. Champion & Co. Ltd. *B.P.* 655,368.

Iron Oxide Pigments. Minnesota Mining & Manufacturing Co., R. C. Johnson, and S. B. Bauman. *U.S.P.* 2,541,068.

Iron ore is treated with sulphuric acid at $> 200°F$. The resultant product containing iron sulphate is rendered plastic and extruded in the shape of cylindrical lumps 1–3 in. long × 0·5 in. in diameter. These lumps are cooled, dried till hard, and then fed into a rotary kiln, where the temperature and time of treatment are controlled so as to decompose the sulphate. C. O. C.

Titanium Dioxide by Vapour-phase Oxidation of Titanium Tetrachloride. du Pont and R. L. Buchanan. *U.S.P.* 2,541,495.

Substituted Amidinomelamines — Flameproofing Agents and Intermediates for Dyes and Auxiliary Agents. American Cyanamid Co., D. W. Kaiser, and B. C. Redmon. (III, p. 382.)

Phthalocyanine Pigment Compositions. Interchemical Corpn. and V. A. Giambalvo. (V, this page.)

Coating Compositions containing γ-Ferric Oxide Hydrate. Columbian Carbon Co., C. D. Downs, and J. Martin. (V, this page.)

Oxidised Ammoniacal Solution of Methylene Resorcinol— Printing or Writing Ink. United States Rubber Co. and J. D. Rourk. (V, p. 392.)

Diazotype Layer containing Three Coupling Components for Neutral Blacks. General Aniline & Film Corpn. and S. C. Slifkin. (IX, p. 395.)

V— PAINTS; ENAMELS; INKS

Photochemical Decolorisation of Mixtures of Berlin Blue and Zinc White. R. Haug. *Farbe und Lack*, **57**, 49–54 (1951): *Chem. Abs.*, **45**, 4463 (25th May 1951).

The fading of paints coloured with mixtures of Paris Blue and Zinc White (I) was considered by Eibner (*Chem. Abs.*, **5**, 3920; **7**, 2313) to be reduction to Berlin White followed by reoxidation. Mixtures of Milori Blue (II) and I in ratios of 1 : 9 to 9 : 1, applied from suspension in glue to drawing paper and exposed to sunlight, filtered sunlight, and ultra-violet radiation (quartz lamp) in a ventilated, glass-covered frame, were bleached when moisture was present by radiation of < 550 mμ., and turned greenish-blue when the colour was restored. Wet slurries containing $> 60\%$ I : $< 40\%$ II were strongly decolorised in a closed system, evolving oxygen, the colour not being restored if the oxygen was removed from the system by white P, Na hydrosulphite, or a mineral-oil barrier. The reaction is thought to be—

$$Fe^{+++}{}_4[Fe(CN)_6]_3 + 2\,ZnO \rightleftharpoons 2\,Fe^{+++}{}_4[Fe(CN)_6] + Zn_2[Fe(CN)_6]^{+}O_2$$

This is confirmed by formation of Berlin Blue from $Zn_2[Fe(CN)_6]$ and $Fe_4[Fe(CN)_6]$ and by suppression of decolorisation of I–II mixtures by addition of $Zn_2[Fe(CN)_6]$. Glycerol, glycol, butanol, acetone, alcohol, and butyl acetate catalysed the reaction, but not CS_2 or naphtha. The decolorisation was retarded but not prevented by low pH (2–3). C. O. C.

PATENTS

Phthalocyanine Pigment Compositions. Interchemical Corpn. and V. A. Giambalvo. *U.S.P.* 2,526,345.

The tendency of phthalocyanine pigments to separate from other pigments with which they are mixed in protective coating compositions is inhibited by replacing 5–40% of the phthalocyanine pigment by its monosulphonic acid or an alkaline-earth metal salt thereof. E. S.

Powdered Colour Base for Surface Coatings. Seidlitz Paint & Varnish Co. and O. Jensen. *U.S.P.* 2,539,429.

A water-insoluble resin is dissolved in a water-soluble solvent, and a pigment ground with the solution until the mixture is homogeneous. It is then slurried with water and filtered, and the finely divided solvent-free pigmented resin obtained is finally dried and powdered. The product is used for colouring paints. C. O. C.

Coating Compositions containing γ-Ferric Oxide Hydrate. Columbian Carbon Co., C. D. Downs, and J. Martin. *U.S.P.* 2,541,642.

γ-Ferric oxide hydrate is particularly adapted for use in coating compositions used in stoved finishes. The orange–yellow pigment upon heating to 300–527°F. in presence of oils or resins becomes a pleasing brown. Its use results in tougher and more resistant films than if α-ferric oxide compounds are used. C. O. C.

Sprayable Coating Composition of High Solids Content. du Pont. *B.P.* 654,446.

A mixture of 10–40-centipoise cellulose nitrate (8–45% on solids present), polymerisable ethers of glycols (42–89%), and a plasticiser (0·5–25·0%) is used. C. O. C.

Zinc Dust–Alkali Silicate Coating Compositions. Industrial Metal Protectives Inc. *B.P.* 653,587.

Zinc dust–alkali silicate coating compositions will keep for long periods of time before setting if a little hydrogen peroxide, lead peroxide, barium peroxide, sodium perborate, potassium chlorate, potassium periodate, manganese dioxide, or sodium persulphate is incorporated in them. C. O. C.

Flatting Agent. U.S. Industrial Chemicals Inc. and K. A. Earhart. *U.S.P.* 2,541,977.

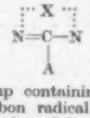
The products obtained by dispersing a Congo resin in aq. alkali-metal hydroxide and then precipitating it by addition of a water-soluble Al, Mg, Ca, Sr, Ba, Zn, or Zr salt while vigorously agitating are flatting agents, which remain stable when compositions containing them are stored or when baked in a film at 250°F. On exposure they retain their original colour and do not darken. C. O. C.

Coating Compositions flatted with Silica Aerogel. du Pont and A. R. Bird. *U.S.P.* 2,541,975.

Addition of approx. 0·1% of alcohol-soluble polyamides, containing as the functional group $-N(CH_2{\cdot}O{\cdot}R){-}CO-$ (R = Alk of < 7 C), to coating compositions, particularly urea–formaldehyde resin compositions, containing finely divided silica aerogel effectively prevents silking, flooding, and settling of the silica and does not adversely affect the cure and other working properties of the composition. C. O. C.

Coating Compositions containing Glyoxalidine Compounds. McCabe Paint & Varnish Co. and H. S. Mannheimer. *U.S.P.* 2,541,825.

Addition of 0·5–5·0% by wt. of a glyoxalidine compound containing at least two groups of formula—



(X = an organic group containing a straight chain of 1–6 C; A = hydrocarbon radical containing a straight chain of 1–6 C; the number of C atoms in the two straight chains is > 9) reduces the surface tension of coating compositions and permits greater penetration of the composition into the surface to be coated. C. O. C.

Mixed Allyl Ethers of Starch— Water-resistant Lacquers. R. M. Hamilton and E. Yanovsky. *B.P.* 653,798.

Mixed allyl ethers of starch containing no free OH groups, e.g. butyl–allyl ether of starch containing 1·5 butyl and 1·5 allyl groups per glucose unit, are soluble in a wide range of organic solvents and produce lacquers of better water resistance than those prepared from allyl starch. They give coatings which after setting polymerise and become insoluble in organic solvents, unaffected by acids or alkalis of moderate concentrations and resisting temp. up to 350°F. C. O. C.

Heat-resistant Aluminium Finish. du Pont and R. A. McClone. *U.S.P.* 2,538,974.

An Al finish which withstands heating up to 1000°F. without failure by charring, flaking, blistering, peeling, discolouring, or embrittling comprises a non-oxidisable resinous vehicle, e.g. a coumarone–indene resin, tricresyl phosphate and ethyl cellulose, Al pigment, and a metal drier, e.g. lead naphthenate. C. O. C.

Water-base Printing Ink. Champion Paper & Fibre Co. and V. A. Lauderman. *U.S.P.* 2,536,555.

An alkaline casein–rosin dispersion of an insoluble pigment in aqueous urea has stability, wetting, spreading, transferring, and dispersible properties comparable with the better grades of oil-base letterpress inks. C. O. C.

Moisture-setting Printing Ink. Sun Chemical Corpn. and J. G. Curado. *U.S.P.* 2,537,513.

Moisture-setting inks containing as binder a β-naphthol pitch containing $< 0{\cdot}1\%$ of glycol-insoluble matter print without fill-in from half-tone plates and neither offset nor smudge after being set. The preferred liquid carrier is 2-methyl-2:4-pentanediol. C. O. C.

Moisture-setting Printing Ink. Sun Chemical Corpn. and J. Hoyt. *U.S.P.* 2,537,531.

A moisture-setting ink of good press stability contains as the binding cellulose nitrate (N content 11·15–11·75%), which is not only freely soluble in the solvent used but is completely soluble in the solvent containing the maximum amount of water present when in equilibrium with an atmosphere of 50–65% R.H. The solvent consists of $>$ 50% of dipropylene glycol and $<$ 50% of a water-immiscible constituent of b.p. $>$ 200°c. The proportions of the two constituents of the solvent are such that it has a hygroscopicity such that a mixture of not $<$ 70 parts of solvent and not $>$ 30 of water is in moisture equilibrium with an atmosphere of 79% R.H. and 20–30°c. C. O. C.

Oxidised Ammoniacal Solution of Methylene Resorcinol—Printing or Writing Ink. United States Rubber Co. and J. D. Rourk. *U.S.P.* 2,540,633.

Oxidation of an aqueous ammoniacal solution of a water-soluble resorcinol–formaldehyde condensate, either by standing for a few days in air or by bubbling air through it for a few hours, yields a dark blue–black, opaque, water-soluble product, which can be used directly as a printing or writing ink depending upon the strength of the solution. The ink flows readily, has good covering power, and dries to a dark brown to black depending upon its strength. It is permanent and does not smear or smudge when wetted. It has good affinity for cellulose and can be used for dyeing or printing textiles. C. O. C.

Hydroxyalkylamides of Butenedioic Acid–Rosin Resins for Steam-set Printing Inks. American Cyanamid Co. and L. E. Cadwell. *U.S.P.* 2,540,776.

A rosin–butenedioic acid adduct is dispersed in an inert organic solvent, and a primary hydroxyalkylamine is slowly added while maintaining a suitable temperature until complete conversion to the salt is obtained. The temperature is then gradually raised to convert the salt to the amide while distilling off the water formed. When the theoretical amount of water has been removed, a volatile organic solvent is added to dissolve the amide resin. The inert solvent is then removed by vacuum distillation to yield a volatile solution of the amide resin. This is used as a vehicle for steam-set printing inks which, while of improved water-tolerance, gives films which on being steamed precipitate the resin in a dry tack-free state. C. O. C.

Printing Ink containing Alkyd Resin and a Mixed Hydrocarbon Solvent. Sun Chemical Corpn. and J. G. Curado. *U.S.P.* 2,541,505.

The preparation of inks which have adequate setting properties is described. C. O. C.

Emulsion Printing Inks. J. M. Huber Corpn. *B.P.* 654,535.

Dispersions of colouring matters in water-in-oil emulsion obtained by use as emulsifying agents of long-chain non-aromatic amines, containing a straight chain of $>$ 14 C and having $>$ 17 C attached to an N atom through carbon bonds, have highly satisfactory flow and printing qualities and yield prints of extreme sharpness. C. O. C.

Invisible Inks. M. S. Kantrowitz and E. J. Gosnell. *U.S.P.* 2,538,784.

A soluble compound of a Group II metal is used. The writing is made visible under ultra-violet radiation by being treated with an alkaline solution of 8-hydroxyquinoline in a polar organic solvent. C. O. C.

VI—FIBRES; YARNS; FABRICS

Clothing an Expanding Population. D. Traill. *J. Textile Inst.*, **42**, P 221–P 236 (June 1951).

World output of wool and cotton is compared with the growth in population, consumption per head of several countries being tabulated. The effect of introducing synthetic fibres is discussed, with emphasis on melt-spun types, e.g. nylon. Preparation of regenerated protein fibres, in particular Ardil, and their application are dealt with at some length. J. W. B.

Cotton Fibre Tensile Strength and X-Ray Orientation. R. Meredith. *J. Textile Inst.*, **42**, T 291–T 299 (July–Aug. 1951).

The tensile strength of single fibres and of flat bundles of fibre for fourteen samples of cotton is correlated with crystallite orientation as measured by X-rays. A correlation coefficient of 0·77–0·84 is found, and is considered inadequate to allow accurate prediction of strength from X-ray orientation. Variation in convolution angle and increasing maturity both affect X-ray orientation but not the strength. Good correlation between X-ray angle and convolution angle suggests that the spiral of the crystallites in all cottons in the original unconvoluted form may be the same. J. W. B.

Strength and Weakness of Cotton Fibres. H. Wakeham and N. Spicer. *Text. Research J.*, **21**, 187–194 (April 1951).

The breaking of cotton fibres has been examined under a polarising microscope, and it is found that breaks usually occur at structural reversals. From studies under a variety of conditions of moisture content, specimen length, and chemical treatment it is concluded that at these regions a change in crystallinity occurs, giving rise to internal stresses which cause the fibre to rupture most easily at these points. When breakage occurs between reversals it is believed to be at structural defects or thin places. The mean breaking stresses for fibres between reversals may be either greater or less than for those breaking at reversals for some varieties of cotton. The distribution of these factors has been discussed for three varieties of cotton. W. J. M.

X-Ray Study of the Transition from Water-cellulose to Cellulose II in Ramie, Jute, and Sisal. R. R. Mukherjee and H. J. Woods. *J. Textile Inst.*, **42**, T 309–T 313 (July–Aug. 1951).

Transition from water-cellulose to cellulose II is measured in ramie, sisal, and jute by drying the fibres progressively. X-Ray photographs of samples, taken over a range of moisture contents, show that in lignified fibres (jute and sisal) the intracrystalline readjustments lag behind those in ramie, as humidity is reduced. This effect is ascribed to the presence of some lignin near the crystallites. Intracrystalline swelling of mercerised ramie, jute, and sisal, as measured by the change in (101) spacing of cellulose II, is found to agree better with the values of Legrand than with those of Hermans and Weidinger. J. W. B.

Conditions for the Formation of Certain Xanthates and the Light Absorption of the Xanthate Group. E. Treiber. *Sitzungsber. Öster. Akad. Wiss.*, **160**, (1–2), 53–62 (1951).

Conditions for the formation of xanthates by addition of CS_2 to an alcohol containing finely powdered KOH have been investigated for sixteen examples. For monohydric alcohols, the facility of reaction follows the esterification rule of Menschutkin and Schwab, viz. primary $>$ secondary $>$ tertiary, although the difference between the first two is slight, whereas the tertiary do not react under the conditions employed. Polyhydric alcohols either do not react or do so incompletely. The main absorption band in each case is due to the C=S chromophore, and the short wave band at $\sim$ 4420 mm.$^{-1}$ to the –S– or –S–CH_3 grouping. H. H. H.

Plasticity of Wool. O. Ripa and J. B. Speakman. *Text. Research J.*, **21**, 215–221 (April 1951).

The slow extension of wool fibres under constant load in distilled water is found to vary considerably in fibres even from the same lock. This creep has been correlated with the sulphur content, and it is suggested that it is due to disulphide-bond breakdown. Chemical treatments confirm this. The activation energy has been found to be about

26 kg. cal., which is consistent with the heat of reaction of 20 kg. cal. for the hydrolysis of cystine with dil. HCl.
W. J. M.

Nature of the Canary Coloration of Wool. J. A. Serra and R. M. A. de Matos. *J. Textile Inst.*, **42**, T 329–T 331 (July–Aug. 1951).

Canary stain is easily reproduced in scoured white wool, either by incubating with other canary-stained wool or by treating in an appropriate buffer solution. The colour of wool incubated at different pH values shows increasing yellowing as the alkalinity increases, and a similar correlation is found between the pH of extracted suint and the colour of raw wool. It is concluded that the canary stain is due to decomposition of cystine residues, and is favoured by high pH of suint and a warm moist climate.
J. W. B.

Structures of Fibrous Proteins. I— A New Polypeptide Structure of α-Keratin. Y. Kanda. *Bull. Chem. Soc. Japan*, **23**, 137–141 (Oct. 1950).

The structure of α-keratin is briefly reviewed, and the effect of steric hindrance discussed. All optically active amino acids from the hydrolysis of natural proteins are of L-type, and a new structure of α-keratin is proposed which can be derived solely from L-amino acids. It is possible to imagine a structure derived solely from D-amino acids, but this is not known in Nature.
W. J. M.

Structures of Fibrous Proteins. II— New Polypeptide Structures of β- and Feather-keratins and Three Silk Fibroins of *Bombyx mori*, *Saturnia pyri*, and *Antheræa mylitta*. Y. Kanda. *Bull. Chem. Soc. Japan*, **23**, 142–146 (Oct. 1950).

New structures for the above proteins are suggested, and it is shown that the calculated identity distance of the unit cells agrees closely with those obtained by X-ray analyses.
W. J. M.

PATENTS

Spinning of Viscose. du Pont. *B.P.* 652,741.

Viscose rayon threads having high tenacity and non-crenelated surfaces are obtained by spinning viscose into aq. H_2SO_4 containing $ZnSO_4$ and 0·5–10 millimoles of a water-soluble organic non-surface-active quaternary ammonium compound (per 100 g. of viscose or bath) in which each of the four organic radicals contains < 5 aliphatic C atoms, at least three of these radicals are completely aliphatic, and the fourth, when aromatic, contains only one benzene nucleus, e.g. benzyltrimethylammonium hydroxide; these quaternary ammonium compounds are effective with both ripened and unripened viscose, and the resulting yarn has high fatigue resistance and improved resistance to soiling and abrasion.
B.P. 652,746.

—Similar improvements in the yarn may be obtained by spinning viscose into acid containing > 2% zinc sulphate and > 0·2% of a soluble aliphatic or *cyclo*aliphatic monoamine modifier containing 4–10 C atoms and no radical of > 6 C atoms, e.g. triethylamine.
W. G. C.

Cellulose Acetate Staple Fibre. British Celanese Ltd., P. F. C. Sowter, and F. B. Hill. *B.P.* 654,900.

Cellulose acetate yarn suitable for conversion into staple fibre is made from a solution of cellulose acetate in acetone by a wet-spinning process in which the coagulating liquid is an aqueous solution of < 38% diethylene glycol diacetate; the yarn is stretched during passage through the coagulating bath, and is then allowed to shrink in a bath of similar composition.
W. G. C.

Rendering Casein Fibres resistant to Hot Dilute Acid. American Enka Corpn. and L. Maaskant. *U.S.P.* 2,539,958.

The fibres are treated with an acid solution of a precondensate of resorcinol or a substituted resorcinol with an aldehyde. The precondensate exhausts from the liquor on to the casein fibres, which are finally dried and baked at 105°c. There is no discoloration of the fibres.
C. O. C.

Formic Acid Solutions of Synthetic Linear Polyamides from α-Amino Acids. du Pont and C. W. Tullock. *U.S.P.* 2,540,855.

Formic acid solutions of α-amino acid polyamides containing at least 20% by weight of –NH·CHR·CO– units (R = methyl, ethyl, *iso*propyl, or *sec.*-butyl) are useful for preparing films and fibres.
C. O. C.

Polyvinylidene Cyanide. B. F. Goodrich Co., R. F. Schmidt, A. E. Ardis, and H. Gilbert. *B.P.* 654,957.

Fibre-forming polymers are obtained by heating monomeric vinylidene cyanide in an ion-free anhydrous organic liquid, e.g. benzene, which is a solvent for the polymer.
W. G. C.

Solvent for Acrylonitrile Polymers and Copolymers. American Viscose Corpn. and M. R. Dalton. *U.S.P.* 2,540,475.

α-Chloro-β-hydroxypropionitrile has a marked solvent action on acrylonitrile polymers and copolymers at 70°c. and completely dissolves them at approx. 100°c., polyacrylonitrile being completely dissolved to a clear solution in 2 hr. at 105°c. The solutions remain stable for indefinite periods at temperatures well below 100°c., no tendency to gel being observed at 50°c. Even below 50°c. gelation takes place very slowly.
C. O. C.

Factors affecting the Service Life of Leather in Wool Processing. R. M. Koppenhoefer. (I, p. 379.)

Thermostable Polyvinylidene Chloride Compositions. Dow Chemical Co. and A. R. Sexton. (XIII, p. 397.)

Identification of *N*-2:4-Dinitrophenylamino Acids. A. G. Lowther. (XIV, p. 400.)

VII— DESIZING; SCOURING; CARBONISING; BLEACHING

Recent Advances in Bleaching and their Significance in British Papermaking. J. Grant. *Proc. Tech. Sect. British Paper & Board Makers' Assocn.*, **32**, 113–141 (Feb. 1951).

The bleaching of wood pulp is reviewed with particular reference to high-density, multi-stage, and chlorine dioxide bleaching of chemical pulp, and sodium peroxide, hydrogen peroxide, and hydrosulphite bleaching of groundwood pulp.
S. V. S.

PATENTS

Continuous Treatment of Vegetable Fibres. Cellulose Development Corpn. Ltd. and F. G. L. Becker. *B.P.* 654,738.

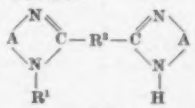
Fibrous vegetable material and treatment liquor, e.g. sodium sulphide or hydroxide, are fed into the top of a cylindrical tower 20–100 ft. high and 5–10 ft. in diameter, the upper part of which may be steam-jacketed. As the material descends it reacts with the solution and forms a compact mass, which is diluted and broken up at the bottom by a cold upward-flowing stream of either water or spent liquor, assisted if necessary by a rotary agitator, and thence drawn off.
J. W. B.

Repressing the Generation of Chlorine Dioxide. Tennants Consolidated Ltd. *B.P.* 655,056.

Water-soluble pyro- and poly-phosphates added to aqueous chlorite solutions at pH 3–7 repress formation of chlorine dioxide. Used with hydrogen peroxide the total effect is greater than the sum of the individual effects. Preferably the weight of phosphate : peroxide is 0·01–8 : 1, and their total concentration should not be > 75 g./litre.
C. O. C.

***N*-Monosubstituted Di-iminazoles— Optical Bleaching Agents.** Ciba Ltd. *B.P.* 653,681.

Compounds of formula—

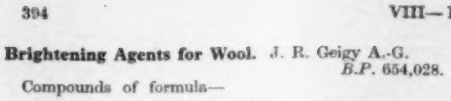


(I)

(A = an aromatic nucleus of the benzene series in which two vicinal C atoms are bound to the two iminazole N atoms; R^1 = subst. or unsubst., Alk or aralkyl; R^2 = an ethylene residue which forms together with the >C:N– double bonds of the iminazole rings an uninterrupted series of conjugated double bonds) are obtained by treating a compound of formula I (R^1 = H) with < 2 mol. of an alkylating or aralkylating agent. They have affinity for cellulose and give a blue to violet fluorescence in daylight and in ultra-violet radiation.
C. O. C.

Brightening Agents for Wool. J. R. Geigy A.-G. *B.P.* 654,028.

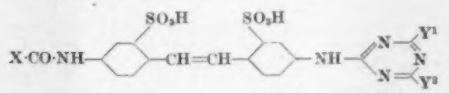
Compounds of formula—



(Y = radical of a 1:3:5-triazine ring which may be substituted in the 3:5-positions by primary, secondary, or tertiary amino groups) have good affinity for wool, blue to bluish-green fluorescence, and very good fastness to light and wet processing. C. O. C.

Brightening Agents. J. R. Geigy A.-G. *B.P.* 654,779.

Compounds of formula—



(X = Alk or alkoxy of 1–6 C; Y^1 and Y^2 each = primary amino or secondary or tertiary amino subst. by Alk, alicyclic, or araliphatic radicals of 1–6 C, or by radicals of the benzene series which may be substituted by non-auxochromes) have affinity for protein fibres from neutral or weakly acid baths. They have good brightening effect and have good fastness to acid, laundering, and sea-water. They are faster to alkalis and exhaust better from neutral baths than 4:5-diphenyliminazolones. C. O. C.

VIII— DYEING

Modern Methods of Fast Dyeing. J. P. Niederhauser. *Teintex*, **16**, 283–297 (June) and 345–359 (July 1951).

A review of newer methods for dyeing cotton, wool, acetate rayon, and nylon, including the following, which are not well known — (1) "Electrofixing" process, developed in Austria and Germany for cotton, mainly for the development of prints but also for the continuous dyeing of vat dyes. It is comparable with the pad–steam process, but in place of steaming the cloth passes through electrically heated metal plates, which permits rapid reduction and fixation of dyes. Development proceeds in the normal manner. (2) Novacel dyes (Nova Chemical Corpn.) are vat dyes applied to a substrate forming a cellulose ether which is soluble in alkali and insol. in water. Cloth is padded in the alkaline soln. of the dye, which is then ppted. on the cloth by means of sulphuric acid. Methyl, ethyl, benzyl, and hydroxyethyl celluloses can be considered, but the last-named is of greatest commercial interest. B. K.

Present and Future Trends in the Dyeing of Woollen Fabrics. A. F. Felgendreger and R. F. Fritz. *Amer. Dyestuff Rep.*, **40**, P 494–P 496, P 504 (6th Aug. 1951).

Progress in dyeing woollen piece goods is broadly discussed under the headings of machinery and equipment, control devices and procedures, new chemicals and dyes, new fibres and new finishes. J. W. B.

Problem of Dyeing Wool in Mixtures with other Fibres. H. Luttringhaus. *Amer. Dyestuff Rep.*, **40**, P 436–P 438 (9th July 1951).

Dyeing of wool–Vicara, wool–nylon, wool–Orlon, A3 staple fibre, and wool–Dacron (–Terylene) is discussed. Dye recipes are given for wool–Vicara blends, together with Munsell notations. Orlon cannot be dyed in presence of wool, and it appears preferable to dye Dacron separately, though some reasonable results are obtained when using monochlorobenzene as a carrier. J. W. B.

Dyeing by the Hot-oil Process. S. H. Williams. *Amer. Dyestuff Rep.*, **40**, P 461–P 468 (23rd July 1951).

Practical vat dyeing temperatures well above 212°F. and a liquor volume amounting to only the pick-up of fabric in an aqueous solution are obtained in a conventional Williams unit by the utilisation of hot mineral oil as the medium in which wet cloth is heated to a high temperature. As reduced dye has negligible solubility in hot oil, bleed-off is prevented, and an equilibrium is attained between dye and fibre. Plant layout and operation are described in detail. J. W. B.

Introduction to Dyeing of Chemstrand Acrylic Fibre. J. A. Woodruff. *Amer. Dyestuff Rep.*, **40**, P 402–P 408 (25th June 1951).

Because of the induced acid-dyeing properties of this modified acrylic fibre, preliminary work is concerned with dyeing those dyes normally used on wool. Effects of such variables as time, temperature, pH, liquor ratio, and Glauber's salt are illustrated with reference to depth of dyeing and penetration. Photomicrographs of cross-sections are included. A low pH of 1·4–1·2 is optimum for exhaustion and penetration, and Glauber's salt is not necessary. Dyeing with acetate rayon dyes and of unions is not well advanced and appears complicated. Bulk dyeing trials suggest the advisability of presetting by wetting out at 200°F., scouring in open width, and dyeing in rope. A brief description is given of finishing techniques, including some to be avoided. J. W. B.

Adsorption Isotherm of Langmuir and of Brunauer, Emmett, and Teller for Multilayers where $n = 3$. D. C. Jones. *J.C.S.*, 1464–1467 (June 1951).

Huttig's Multilayer Isotherm. R. M. Barrer. *J.C.S.*, 1874–1876 (July 1951).

PATENTS

Pad Dyeing with Insoluble Pigments. Sherwin-Williams Co. and J. M. Kuhn. *U.S.P.* 2,539,914.

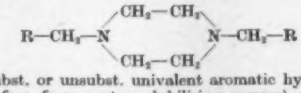
A pigmented solution of a lacquer in an organic solvent to which has been added two or more binders each separately soluble in the solvent but mutually incompatible therein is readily emulsifiable in water to give a lacquer-in-water emulsion, which remains stable under the most severe conditions of pad dyeing. Cloth dyed with such an emulsion after the usual soaping and finishing has an excellent handle and is fast to washing, dry cleaning, rubbing, and light. The preferred binders are ethyl cellulose (1–4 parts by weight) and an organic-solvent-soluble urea– or melamine–formaldehyde resin (4). C. O. C.

Improving the Fastness of Vat-dyed Nylon. du Pont and W. H. Sharkey. *U.S.P.* 2,541,839.

Vat-dyed nylon containing the dye as particles of 0·2–5·0 μ. is fast to light, washing, and particularly rubbing. The dye particles may be obtained of this size by heating the vat-dyed nylon in an aq. soln. of an aliphatic polyhydroxy compound containing 2–6 C and 2–3 alcoholic hydroxyls, preferably ethylene glycol. Another method is to heat the moistened vat-dyed nylon intermittently in mineral oil at 110–210°C. C. O. C.

Gas-fading Inhibitors— 1:4-Bisarylmethylpiperazines. General Aniline & Film Corpn., H. W. Grimmel, and A. Guenther. *B.P.* 654,788.

Compounds of formula—



(R = subst. or unsubst. univalent aromatic hydrocarbon radical free from water-solubilising groups) are useful gas-fading inhibitors for application in the dyebath. They are fast to light, dry cleaning, and laundering. C. O. C.

Gas-fading Inhibitors. General Aniline & Film Corpn., A. F. Strobel, and W. W. Williams. *U.S.P.* 2,539,212.

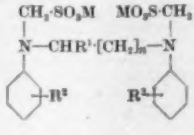
Compounds of formula—

$$R^1{\cdot}CH_2{\cdot}NR^2{\cdot}CH_2{\cdot}CH_2{\cdot}NR^2{\cdot}CH_2{\cdot}R^1$$

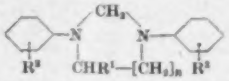
(R^1 = subst. or unsubst. phenyl; R^2 = Alk, alkylene, alkoxyalkyl, cyanoalkyl, or carboxyalkyl, but must contain no halogen), e.g. *NN'*-dibenzyl-*NN'*-diethylethylenediamine, are gas-fading inhibitors which may be used in the dyebath. They are fast to dry and wet cleaning and sublimation, and may be used with pastel dyeings as they form no colour when exposed to gas fumes. C. O. C.

Gas-fading Inhibitors—*NN'*-Bissulphomethyl-*NN'*-diarylalkylenediamines. du Pont, D. E. Kvalnes, and C. W. Croco. *U.S.P.* 2,541,822.

Compounds of formula—



(R^1 and R^2 = H or CH_3, n = 1 or 2, M = a cation which does not impart solubility in water), obtained by treating the formaldehyde-bisulphite compound of the cation with a diarylalkylenediamine in aqueous alcohol, form hydrates which dissolve readily in cold water. Used in the dyebath these hydrates gradually decompose on heating to form on the fibre water-insoluble compounds of formula—



which impart excellent resistance to gas fading. C. O. C.

Colouring Cloth with a Pigment dispersed in Lacquer. Interchemical Corpn. and L. P. Hill. *U.S.P.* 2,540,048.

Cloth is dyed by impregnating it with an aqueous emulsion of a pigmented lacquer, containing as binder a resin which on heating becomes insoluble in organic solvents, and then heat-curing. Unless a stiff finish is desired the emulsion used should not contain > 2·5% of resin. C. O. C.

Evenly Dyeing Carrotted Fur Fibres. Hat Corpn. of America and S. G. Hoffman. *U.S.P.* 2,539,907.

Even dyeings showing no mottling are obtained on fabrics of carrotted fur fibres by use of a mixture of an acid dye and a premetallised acid dye. Acid dyes have less affinity for the tips of carrotted fibres than for the other portion, whereas the reverse is true for premetallised dyes. The proportion of each class of dye to be used depends on the degree of carrotting of the fibres. C. O. C.

Imparting a Bright Red to Meat and other Materials containing Oxygen-bearing Pigments. Armour & Co., H. M. Coleman, and A. H. Steffen. *U.S.P.* 2,541,572.

Meat, etc. is turned bright red by treating it with ascorbic acid. C. O. C.

Colouring Anodised Magnesium and its Alloys. Consolidated Vultee Aircraft Corpn. and P. R. Cutter. *U.S.P.* 2,538,831.

The metal is made the anode in an electrolytic cell containing as electrolyte a solution of a silicate, a caustic alkali, and if desired an organic compound, e.g. phenol. D.C. of 10–20 amp./sq. ft. is used for 10–30 min. or A.C. of 60 cycles and 20–30 amp./sq. ft. The anodised metal is mordanted in an aq. soln. of 0·1–0·4% of ammonium acetate and 0·1–0·6% of a metal acetate at 180–190°F. for 1 min. It is then dyed preferably with an acid or chrome dye using approx. 2 g. dye/litre and a liquor at pH 5·0–5·5 and 65–80°C. The dyeings are very fast to light. C. O. C.

Fluorescent Resins for rendering Textiles Crease- and Shrink-resistant. Sun Chemical Corpn. and R. C. Ackerman. (III, p. 382.)

Oxidised Ammoniacal Solution of Methylene Resorcinol—Printing or Writing Ink. United States Rubber Co. and J. D. Rourk. (V, p. 392.)

IX—PRINTING

Origin of Calico Printing in Switzerland with a Glance at the Technique of Printing in the Eighteenth Century. R. Haller. *Textil-Rund.*, **6**, 299–315 (July 1951).

Printing of Synthetics—Trends and Mill Problems. A. F. McLean. *Amer. Dyestuff Rep.*, **40**, P 501–P 503 (6th Aug. 1951).

Modern technique and problems of colour printing are discussed. J. W. B.

Diazotype Layer containing Three Coupling Components for Neutral Blacks. General Aniline & Film Corpn. and S. C. Slifkin. *U.S.P.* 2,537,919.

Blacks which are uniformly neutral throughout the half-tones and full-density areas are obtained by using a light-sensitive diazo compound derived from a *p*-diamine of the benzene series and three coupling components. One coupling component has the formula $R^1{\cdot}CO{\cdot}CH_2{\cdot}CO{\cdot}NR^2R^3$ (R^1 = Ar, Alk, or aralkyl; R^2 and R^3 = same or different, H, Ar, Alk, aralkyl, acyl, or hydrogenated cyclic radical). Another, which must have a different coupling potential from the first, is of formula $HO{\cdot}R^4{\cdot}NH{\cdot}X$ (R^4 = a naphthalene radical with no substituent *ortho* or *para* to the OH group; X = residue of a $-C(:NH){\cdot}NH_2$ or $-C(:NH){\cdot}NH{\cdot}C(:NH){\cdot}NH_2$ group) or $HO(2){-}R^5{-}(3)OY(1)$ (R^5 = sulphonated naphthalene radical; Y = H, hydroxyalkyl, carboxyalkyl, or aminoalkyl). The third coupling component has formula I but contains no nuclear sulphonic acid group when the second component has also formula I. The molar ratio of the third to the first coupler should be 0·5–1. C. O. C.

PATENTS

Photographic Printing of Textiles. General Aniline & Film Corpn. and S. C. Slifkin. *U.S.P.* 2,541,178.

Textiles of any kind are impregnated with a diazonium compound of an aromatic amine decomposable by light to form a non-coupling compound, an organic compound coupling in alkaline medium, a precoupling preventive, and zinc chloride. They are then exposed to light through a stencil or photographic negative, and finally developed by suitable alkaline treatment. Positive images showing excellent gradation and density are obtained. C. O. C.

Photomechanical Printing. E. Zieger and L. Frank. *B.P.* 655,016.

Continuous boundary lines for each part of the design and satisfactory reproduction of any fine lines in the design are obtained by placing the design over a light-sensitive surface and making two exposures, one to diffused light and one to a concentrated unidirectional beam of light. The negative is then developed and dried, but not fixed until it has been once again exposed and developed. The last exposure is made without the original design but if desired with use of a screen or any shading medium to suit the nature of the design. C. O. C.

Deposition of Material according to a Pattern. Electric & Musical Industries Ltd. and R. E. Spencer. *B.P.* 655,032.

The desired pattern is formed as an electrically conductive layer, and the material to be deposited is attracted towards the pattern by electrically charging the conductive layer. C. O. C.

Colour Photography. S. Papadakis. *B.P.* 655,344.

Three negatives are made using a tricolour selective device, and from these three positives are made. One positive is coloured to form a monochrome, and then the two other colours are applied to it. Each colour is applied by first sensitising with dichromate, then exposing through another positive, and washing and colouring with the corresponding dye. Acid dyes are particularly suitable for use with this process, e.g. *C.I.* 520, 1239, and 10 for blue, red, and yellow respectively. C. O. C.

Multicolour Diazotype Layers. General Aniline & Film Corpn. and S. C. Slifkin. *U.S.P.* 2,542,715.

A light-sensitive monodiazo derivative of a *p*-diamine of the benzene series is used with an acetoacetylarylamide as a fast coupling component and a substituted mono- or dihydroxynaphthalene compound as a slow coupler, the proportions of the couplers being 1 : 10–35 by weight. This gives images in which the shadows and highlights of the original are reproduced in different colours, thus indicating the difference in densities without need for creating such a low density in the highlights as to render them indistinct. *U.S.P.* 2,542,716.

The fast coupler is 2:3-dihydroxynaphthalene, 1:8-dihydroxynaphthalene-4-sulphonic acid, phenylmethylpyrazolone, or orcinol; and the slow coupler *m*-hydroxyacetanilide, *m*-hydroxyphenylurea, 7-hydroxynaphthalene-1:2-iminazole, ethylenediamine amide of 2-hydroxy-3-naphthoic acid, or 7-hydroxynaphthalene-1-diguanide, the proportions of the couplers being 1 : 10–40 by weight. C. O. C.

***N*-Substituted *iso*Alloxazines as Catalysts in the Dye Bleach Baths for Colour Photography.** General Aniline & Film Corpn. and F. W. H. Mueller. *U.S.P.* 2,541,884.

Transfer Printing on to Curved Surfaces. V. J. Tenety. *U.S.P.* 2,542,083.

Oxidised Ammoniacal Solution of Methylene Resorcinol—Printing or Writing Ink. United States Rubber Co. and J. D. Rourk. (V, p. 392.)

X—SIZING AND FINISHING

Trends in the Chemical Processing of Cotton Fabrics. W. M. Scott. *Amer. Dyestuff Rep.*, **40**, P 473–P 476 (23rd July 1951).

A brief survey is made of the history of finishing and of the chemistry of recent modifications. Recent developments of partial acetylation, carboxymethylation, phosphorylation, aminisation, and treatment with carboxymethyl cellulose are discussed in more detail. J. W. B.

Titanium as an Agent for imparting Durable Fire-retardancy to Fabrics. A. E. Jacobsen, W. F. Sullivan, and I. M. Panik. *Amer. Dyestuff Rep.*, **40**, P 439–P 443 (9th July 1951).

Various types of fire-retardant treatments are classified as non-durable (water-soluble salts), semi-durable (nitrogenated phosphates), and durable (organic and inorganic). Studies on the inorganic titanium–antimony systems are extended to cover the introduction of silicates in the neutralisation bath to minimise afterglow. Fabric is treated with titanium chloride–acetate solution, partly dried, and aftertreated in a sodium carbonate bath containing 20 g. of sodium silicate per litre. Time of afterglow is greatly reduced, the effect being fast to prolonged laundering. A mechanism for flame retardance is suggested. J. W. B.

PATENTS

Treating Hydrogen-donor Textiles with *iso*Cyanate Composition— Rendering Wool Goods Shrink-resistant. American Cyanamid Co., E. L. Kropa, and A. S. Nyquist. *U.S.P.* 2,537,064.

Hydrogen-donor textiles, e.g. wool and regenerated cellulose, are treated with a mixture of (1) a compound of formula $CH_2{:}CR{\cdot}A_n{\cdot}CH_2{\cdot}NCO$ (R = H or CH_3; A = bivalent sat. alkylene or arylene which may be substituted with Ar or Alk respectively; $n = 0$ or 1, the CH_2 group attached to the NCO group being attached directly when $n = 1$ to any C atom of A), and (2) another compound copolymerisable with (1), containing a single $CH_2{:}C<$ group, and free from H reactive with the NCO group. The proportions of the two ingredients are such that on curing the resultant polymer contains not < 2 NCO groups per mol. The material is then heat-cured. The process is particularly suitable for reducing the felting power of wool, e.g. by impregnating with 3–15% by weight of a copolymer of allyl *iso*cyanate (5–20% by weight) and ethyl acrylate (95–80%). C. O. C.

Insect-repellent. U.S. Secretary of the Army, P. D. Bartlett, and A. Schneider. *U.S.P.* 2,537,022.

Fabric impregnated with a compound of formula—

$$CH_2{\cdot}[CH_2]_n{\cdot}C(C_2H_5)(CH_2{\cdot}CH_2A){\cdot}CHO$$

(A = CN or COOH; $n = 1$–3), e.g. 2-ethyl-2-β-cyanoethylbutyraldehyde, is rendered repellent to mosquitoes and other insects.

U.S.P. 2,537,023.

Fabric impregnated with indanol, acetoxyindanol, and methoxyindanol is insect-repellent. Thus fabric treated with α-indanol repelled *Aedes ægypti* for 1–5 days; that treated with β-indanol was repellent for at least 15 days. C. O. C.

Cellulosic Textile Shrinkage Control and Crease Resistance with Inhibited Tendering Action. Alrose Chemical Co. and L. Beer. *U.S.P.* 2,541,457.

When treating cellulosic textiles with aqueous solutions of aliphatic aldehydes, e.g. formaldehyde or glyoxal, in order to obtain crease- or shrink-resistance, addition of an alkali-metal sulphate greatly inhibits the liability to tendering by the acid catalyst used. C. O. C.

Felted Cotton Material. Kendall Co. *B.P.* 654,834.

A strong and flexible cotton felt is produced by subjecting a body composed wholly or mostly of cotton fibres to the action of a strongly alkaline swelling agent for cellulose, e.g. 8–30% caustic soda, while supporting it without tensional stress, thus causing intercurling or kinking of the fibres and contraction of the body area, after which the swelling agent is removed, again without stretching, thus fixing the interlocked fibres.

B.P. 654,869.

A non-woven, felt-like material which is soft, porous, flexible, conformable, compressible, and substantially extensible is formed from a carded cotton web by treating it with caustic soda to cause interlocking of the fibres by curling and kinking. J. W. B.

Reducing the Felting or Shrinking Properties of Material containing Wool. Tootal Broadhurst Lee Co. Ltd. and R. P. Foulds. *B.P.* 654,739.

Wool can be rendered resistant to felting by leaving it for a time in contact with a solid substance capable of liberating an oxidising or reducing gas. Thus in one example an unscoured knitted all-wool fabric at normal regain was covered with 1·5 times its own weight of bleaching powder. It was then rolled up and left for 6–8 hr. at room temperature, and the powder then removed by shaking and brushing in a current of air. The fabric was then passed through an aqueous bath containing Calsolene Oil (ICI), thoroughly washed in cold water, treated in 1% aq. $NaHSO_3$, rinsed, and dried. C. O. C.

Rendering Wool Resistant to Felting during Wet Processing. Geo. W. Bollman & Co. Inc. and M. J. Reider. *U.S.P.* 2,541,780.

Adsorption on the surface of the fibre of a material of particle size such as to clog the fibre scale renders wool resistant to felting. Any water-insoluble material of particle size not > 1 μ. may be used, 5–20% by weight being necessary and application being from an aqueous dispersion. If the material carries a negative charge, e.g. bentonite, Fuller's earth, or silica, then the suspension should be acid, preferably at pH 2·0–3·5, and subsequent wet processing should be acidic; if the material is positively charged, e.g. base-exchange resins, then it should be applied from a dispersion at pH 8·5–11·0, and subsequent wet processing must be alkaline. To remove the material the wool is rinsed under the opposite (alkaline or acid) conditions to reverse the polarity of the wool from that at which the material was applied. C. O. C.

Dimensional Stabilisation of Wool-containing Materials. American Cyanamid Co., L. A. Fluck, Jr., J. E. Lynn, and E. A. Schuman. *U.S.P.* 2,539,365.

In the dimensional stabilisation of wool-containing material by treatment with aminoplasts, considerable reduction in the amount of aminoplast necessary is effected by treating the material with an alkaline solution of a peroxy compound before the resin treatment. Dyeing may be carried out between the peroxy and the resin treatments.

American Cyanamid Co., L. A. Fluck, Jr., J. E. Lynn, and E. L. McPhee. *U.S.P.* 2,539,366.

The material is given the peroxy treatment, then treated with an aqueous colloidal solution of a cationic aminoplast, and finally heat-cured. C. O. C.

Treating Heat-set, Oriented Nylon Fabric with Formaldehyde. du Pont, B. Graham, and O. E. Schupp, Jr. *U.S.P.* 2,540,726.

The heat-set fabric is impregnated with a catalyst of pH < 3, dried at 100–250°c., and while maintained at fixed dimensions heated with anhydrous formaldehyde vapour at 100–250°c. until the nylon contains at least 0·5%, preferably $> 1·5$%, by weight of formaldehyde. This imparts liveliness, fullness, crease and solvent resistance, high m.p., and increased affinity for dyes. C. O. C.

Mothproofing from Dry Cleaners' Solvent. Merck & Co. Inc. and E. A. Wolff. (III, p. 383.)

XI—PAPER AND OTHER CELLULOSIC PRODUCTS

Mechanism of the Hydrolytic Cleavage of the Inter-unit Bonds in the Cellulose Molecule. G. Beall and L. Jörgensen. *Text. Research J.*, **21**, 203–214 (April 1951).

The use of statistical moments is described as applied to the problem of the degradation of cellulose by hydrolysis. The results are confirmed experimentally and substantiate that hydrolytic cleavage occurs at random. The method avoids making any restriction on the nature of the starting material. W. J. M.

Absorption Spectra of some Cellulose Derivatives in the 3-μ. Region. L. Brown, P. Holliday, and I. F. Trotter. *J.C.S.*, 1532–1539 (June 1951).

The infra-red absorption spectra of native and regenerated cellulose, and of the cellulose cyanoethyl

ethers, ethyl ethers, acetates (primary and secondary), acetate–butyrates, laurate, laurate–naphthenate, and acetate–stearate, have been measured in the region 2·5–4·0 μ. The results show that the shape and frequency of the absorption band associated with the fundamental hydroxyl stretching mode give considerable information about the hydrogen-bonding conditions, and in particular may be used as a criterion of uniformity of substitution along the cellulose chains. The possibility of using the intensity of the hydroxyl band as a measure of the hydroxyl content of cellulose derivatives is discussed. Preliminary infra-red measurements are described on the water imbibition of some cellulose ethers and esters.

H. H. H.

New Data on the Chemical Composition of Oxycelluloses. E. D. Kaverzneva. *Doklady Akad. Nauk S.S.S.R.*, **78**, 481–483 (21st May 1951).

Previous work (cf. *Chem. Abs.*, **44**, 1257 (1950)), indicating the presence of ketone groups in various oxycelluloses, is now confirmed, and evidence is advanced concerning the position of the ketone group in the glucose residue. Oxycellulose, made by hypochlorite oxidation, was first treated with sodium hypoiodite, to convert CHO groups to COOH, and then condensed with hydroxylamine. The product was reduced by metallic calcium in aqueous medium and then hydrolysed to the monomer by acid. Primary amines were shown to be present in the hydrolysate, thus confirming the presence of ketone groups in the original oxycellulose. Tests on the hydrolysate for glucosamine (ketone group in 2-position) were weakly positive. It was found also that oxycelluloses of various types give strong positive phosphomolybdate reactions for the presence of –C(OH):C(OH)– groupings, which are formed from –CH(OH)-CO– groupings at pH > 10. Cellulose oxidised with periodic acid does not give this reaction. It was found also that, when hydroxylamine reacts with oxycelluloses other than those prepared by oxidation with periodic acid, hydroxamic acids are formed as well as oximes. It is considered that the 6-carboxyl groups present in these oxycelluloses may form lactones, probably with 3-hydroxyls, and that hydroxylamine reacts with these to give hydroxamic acids. The presence of such lactone bridges explains why an oxycellulose may have a "total" carboxyl content, determined alkalimetrically, that is less than its uronic carboxyl content, determined from the wt. of CO_2 liberated on boiling with acid.

A. E. S.

PATENTS

Paper Size. P. H. Glatfelter Co. and A. S. Erspamer. *U.S.P.* 2,541,692.

A mixture of rosin and an ester of glycerol and maleic anhydride of acid number not < 250 gives very uniform sizing and does not lower the quality of the paper, and there is no limit to the degree of sizing that can be obtained. C. O. C.

Coating or Impregnating Paper Stock, Loose Cotton, etc. with Wax, Resins, Rubber, etc. American Cyanamid Co., J. H. Daniel, Jr., L. H. Wilson, R. Hastings, and C. G. Landes. *B.P.* 654,955.

The material is suspended in water, and then an acid condensate of a urea–formaldehyde condensate with a cationic water-soluble polyamino compound having an H atom attached to an amino N atom is added. An aqueous dispersion of the wax, resin, etc. in deflocculated condition is added, when the cationic resin present causes the wax or the like to be deposited uniformly on the cellulosic fibres. The treated fibres even when coated with more than their own weight of wax or the like can still be formed into sheets, while loss of the impregnating compound in the white water system is largely avoided. C. O. C.

Paper of Improved Permanence and Stability on Storing. American Cyanamid Co., J. Studeny, J. D. Pollard, and C. G. Landes. *U.S.P.* 2,539,558.

Incorporation of 0·3–10·0% by weight of dicyandiamide or its lower alkyl or hydroxyalkyl substitution products effectively stabilises paper to deterioration on storing. C. O. C.

Wet-strength Paper. Munising Paper Co. and W. A. Schenck. *U.S.P.* 2,540,352.

Paper impregnated with a synthetic linear polyamide, formaldehyde, and an acid catalyst, dried, washed free from acid, and again dried is highly absorptive, and its wet and dry tensile strength and resistance to linting and rubbing are improved. C. O. C.

Carbon Paper. F. G. Francis. *B.P.* 653,573.

Spirit reproducing carbon paper is rendered clean to handling by coating the transfer layer with varnish containing a plasticiser and a wax, the latter causing the varnish layer to be readily broken by a type head. C. O. C.

Rubberised Abaca Fibre Paper. W. A. Hermanson. *U.S.P.* 2,541,763.

A base sheet of pure, unused abaca (*Musa textilis*) fibres having interfibre interstices of 5–150 μ., the fibres being 4–7 mm. long and 17–25 μ. in diameter, when impregnated with a suitable natural or synthetic latex yields a sheet material of high strength not hitherto attainable. A suitable impregnating liquor consists of latex of 40–50% solids (50 parts), solution of a fluorescent dye (stilbene derivative, triazine, or modified umbelliferone) 5 grains per 100 ml. water (45), and glycerol (5), the take-up being such as to give a weighting of 165% on drying. C. O. C.

Continuous Films of Regenerated Cellulose. Svenska Rayon A.-B. and C. A. J. Lindqvist. *B.P.* 654,783.

Viscose is extruded from a pair of separate nozzles into coagulating liquid under conditions to coagulate into a film or sheet from each nozzle, both sides of each sheet being exposed until non-tacky. The sheets are then put together, and further treatment is applied to the outer faces only, finally separating and drying. More even penetration is effected, leading to a uniform product, and the sheets reinforce each other during treatment, preventing breakage. J. W. B.

Colour-stable Cellulose Ethers. Celanese Corpn. of America and W. B. Horback. *U.S.P.* 2,541,252.

Addition of 3% by weight of a neutral organic phosphite imparts complete colour-stability to cellulose ether compositions. C. O. C.

Recent Advances in Bleaching and their Significance in British Papermaking. J. Grant. (VII, p. 393.)

XII—LEATHER; FURS; OTHER PROTEIN MATERIALS

PATENT

Tanning Hides or Skins. J. S. F. Aabye. *B.P.* 655,094.

The skins are partly tanned by any method and then frozen and dried by subliming the ice from them under sub-freezing conditions. The products have the same characteristics as normally tanned skins, even if such a small quantity of tanning agent has been used that normally no tanning effect would be obtained. C. O. C.

XIII—RUBBER; RESINS; PLASTICS

PATENTS

Vulcanised Synthetic or Natural Rubber containing Rutile Titanium Dioxide. Spolek pro Chemickou a Hutni Vyrobu N.P. *B.P.* 653,814.

Addition of rutile TiO_2 to rubber mixes improves both the lightness of the colour of the products made from them and their mechanical properties. In addition less vulcanisation accelerator is needed, and the products are considerably more resistant to ageing than those produced with anatase TiO_2. C. O. C.

Vulcanisation Retarders. Monsanto Chemicals Ltd. and W. M. Morgan. *B.P.* 653,538.

Metal (except alkali-metal) salts of organic sulphonic acids, e.g. zinc dodecylbenzenesulphonate, are vulcanisation retarders which are particularly useful in presence of furnace black.

B.P. 653,559.

An organic hydrogen sulphate or its metal (other than alkali-metal) salt, e.g. zinc lauryl sulphate, is used. C. O. C.

Thermostable Polyvinylidene Chloride Compositions. Dow Chemical Co. and A. R. Sexton. *U.S.P.* 2,537,713.

Incorporation in polyvinylidene chloride compositions of 5–12% by weight of α-methylbenzyl benzoate imparts stability to heat. C. O. C.

Polyvinyl Chloride Composition heat-stabilised with a Mixture of Monoalkoxyethyl Fumarate or Maleate and a Basic Lead Salt. Monsanto Chemical Co. and J. R. Darby. *U.S.P.* 2,539,362.

Heat-stabilised Polyvinyl Chloride Compositions. Monsanto Chemical Co. and G. R. Buchanan, Jr. *U.S.P.* 2,542,179.

Addition of a basic heat stabiliser, particularly a basic lead salt, together with cinnamic acid to polyvinyl chloride compositions gives better thermal stability than the use of the basic heat stabiliser alone. C. O. C.

Polymerised Haloprene Compositions. du Pont. *B.P.* 654,689.

Products of outstanding abrasion resistance, useful for printers' blankets, tyres, floor covering, etc., are formed by mixing polymerised 2-halogeno-1:3-butadiene and halogenated polyethylene cured above 100°c. In one example the composition is— 2-chloro-1:3-butadiene plastic polymer (9·0 parts by wt.), chlorinated polyethylene containing 55% Cl (4·5), sulphur (0·072), magnesium oxide (0·9), phenyl-β-naphthylamine (0·072), stearic acid (0·144), coumarone resin (0·6), calcium carbonate (9·0), carbon black (11·2), pine tar (0·6), zinc oxide (0·9), and N.W. rosin (0·6). After rolling into a sheet, or applying from a solvent to cotton fabric, the material is vulcanised or cured. J. W. B.

Copolymer Latices. B. F. Goodrich Co. and E. B. Osborne. *B.P.* 653,359.

Copolymer latices are prepared by emulsion copolymerisation of vinyl chloride (75–90%), vinylidene chloride (5–15%), and an alkyl acrylate (5–15%) in which the two latter are added gradually to an emulsion of the former during polymerisation. The products are latices of high stability, which on evaporation deposit transparent elastic films of high strength and low permeability to water, oils, and greases, and show good adhesion to paper, wood, and porous materials. E. C.

Acrylonitrile–*iso*Butylene Copolymers. American Viscose Corpn. and M. R. Lytton. *U.S.P.* 2,537,146.

Copolymers of not $<$ 65% of acrylonitrile and not $<$ 35% of *iso*butylene are soluble in acetonitrile or a nitroalkane. Such solutions yield films and coatings which are dimensionally stable, not shrinking to any appreciable extent even when treated with boiling water, tough, and resilient, impervious to acids and alkalis, and resistant to weathering. C. O. C.

Solvent for Polyacrylonitrile. American Viscose Corpn. *B.P.* 654,784.

A mixture of nitromethane (60–80% by vol.) and α-hydroxypropionitrile (40–20%) is not a swelling agent for polyacrylonitrile at room temperature but completely dissolves it at 80–90°c. to form colourless, homogeneous solutions. C. O. C.

Modifying Polymers by Treatment with Ethylene. du Pont, W. E. Hanford, and J. R. Roland. *U.S.P.* 2,542,771.

Natural and synthetic polymers may be modified by treating them under pressure with ethylene in presence of a compound producing free radicals as catalyst. The effect of the treatment varies; thus cellulose esters can be rendered less compatible with organic solvents, or the saponification value of vinyl ester polymers can be reduced. C. O. C.

Cross-linked Polymers. du Pont and P. W. Morgan. *U.S.P.* 2,539,920.

Polymers containing at least one thiocyano-ester, halogeno-ester, sulphate ester, or sulphonate ester group can be directly cross-linked by heating them at not $<$ 50°c. with 5% by weight of a polythiourea compound containing the >N·CS·NH– group. C. O. C.

***N*-Substituted Triazines and Synthetic Resins therefrom.** British Industrial Plastics Ltd., J. C. Bacon, and W. F. Hart. *B.P.* 653,827.

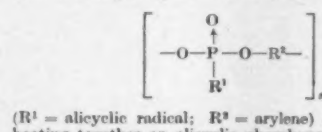
The products obtained by heating a monochlorodiaminotriazine (in which one or both amino groups may be alkylated) or a dichlorodiaminotriazine (whose amino group may be alkylated) with a drying-oil amine in presence of an alkaline agent when treated with formaldehyde yield air-drying, thermosetting resins. These resins have not the brittleness of straight unmodified thermosetting amino resins nor the softness of unmodified alkyd resins, and unlike the latter are insensitive to alkalis. They are useful in preparing paint, lacquer, and varnish vehicles, and for rendering cloth or paper water-repellent. C. O. C.

Polymeric 4-Vinylcyclohexene Dioxide. Canadian Industries Ltd. *B.P.* 654,730.

The compositions obtained by dissolving a polyvinylacetal in 4-vinylcyclohexene dioxide and then polymerising the latter in presence of a catalyst are particularly useful in protective coatings. They may be coloured by adding a suitable dye to them before polymerising. C. O. C.

Polyphosphonate Resins. I.C.I. Ltd., H. Zenftman, and R. McGillivray. *B.P.* 653,489.

Polyphosphonate resins of good heat resistance and low inflammability, of general formula—



(R^1 = alicyclic radical; R^2 = arylene) are prepared by heating together an alicyclic phosphonyl dichloride, e.g. *cyclo*hexylphosphonyl dichloride, and a dihydric phenol, e.g. *pp'*-dihydroxydiphenyl, if desired in presence of a metal catalyst, e.g. tin. E. C.

Rapid-drying Coating Composition. Standard Register Co., F. M. Fryman, and N. Sugarman. *U.S.P.* 2,536,107.

The liquid precondensate of a furan-type aldehyde (2–4 parts) and an arylenediamine, with or without other film-forming materials, yields films which dry in 30 sec. or less. It is particularly suitable for preparing carbon paper. C. O. C.

Wrinkle Coating Composition. New Wrinkle Inc. and W. F. Flanagan. *U.S.P.* 2,536,049.

Films having a pattern of smooth and wrinkled areas are obtained with only one coating composition by heating the areas intended to be wrinkled until a skin is just formed and then immediately applying water. The remaining areas are heated either less than is necessary for skin formation or until any skin formed has been rendered unaffected by water. Preferably the films are formed and treated on a glass plate, from which they are removed for use. C. O. C.

Multivalent Metal Pectinate Films. U.S. Secretary of Agriculture, H. S. Owens, and T. H. Schultz. (III, p. 383.)

XIV—ANALYSIS; TESTING; APPARATUS

Microdetermination of Nitrogen as Ammonia by the Diffusion Technique. H. Naylor. *J. Textile Inst.*, 42, T 300–T 308 (July–Aug. 1951).

The diffusion technique is investigated with a view to the analysis of small textile samples containing relatively large proportions of nitrogen, e.g. protein fibres, nylon, or cellulosic materials containing urea– or melamine–formaldehyde resins. Standard Conway units give erratic results, but a deeper form made from polystyrene or Perspex (ICI) proves satisfactory. Kjeldahl digests are diluted, and aliquot microportions transferred to the outer annular cell; standard NaOH is added. The inner circular compartment contains standard H_2SO_4, which is titrated with a microburette after 18 hr., during which time the cell is sealed with a Pyrex glass lid. Tabulated data include recoveries of ammonia from ammonium sulphate and determination of nitrogen in degummed silk and nylon. J. W. B.

Substituted Benzidines and Related Compounds as Reagents in Analytical Chemistry— VI and VII. *J.C.S.*, 1516–1521 (June 1951).

VI— Precipitation of Tungsten. R. Belcher and A. J. Nutten. Pp. 1516–1520.

VII— 3:3'-Dimethylnaphthidine as Indicator in the Titration of Zinc with Ferrocyanide. R. Belcher, A. J. Nutten, and W. I. Stephen. Pp. 1520–1521.

Examination of Detergent Preparations. Society of Public Analysts and Other Analytical Chemists, Analytical Methods Committee. *Analyst*, **76**, 279–286 (May 1951).

Report of a subcommittee set up to consider whether standard methods of analysis of soapless detergents were possible. It is concluded that a method of quantitative determination suitable for all types is impracticable. A method is described for the qualitative examination of an unknown composition, to ascertain the type of detergent present, and detailed descriptions are given of quantitative methods which have proved satisfactory for the determination of— total organic matter, anionic detergents, sulphated bodies, sulphonated bodies, non-ionic detergents, cationic detergents, and neutral and alkaline electrolyte. W. K. R.

Titration of Anion-active Colloidal Electrolytes with *p*-Toluidine. H. Stüpel and A. von Segesser. *Helv. Chim. Acta*, **34**, 1362–1368 (Aug. 1951).

The most important of the anion-active colloidal electrolytes employed in the detergent industry may be quantitatively determined by complex formation with *p*-toluidine hydrochloride, followed by extraction and titration with standard NaOH using *o*-cresolsulphophthalein as indicator. Alkarylsulphonates, secondary alkylsulphonates, and secondary alkyl sulphates require CCl_4 as solvent and 0·1 N-NaOH; primary alkyl sulphates and fatty acid condensation products need ether for extraction; and alkali-metal carboxylate soaps may be quantitatively titrated with N. alkali with CCl_4 as phase separator. The method is particularly suitable for series analyses. H. H. H.

Analysis of Organosilicon Compounds— Qualitative Analysis of Alkyl- and Aryl-chlorosilanes, Alkyl- and Aryl-alkoxysilanes, Tetra-alkylsilanes, and other Organosilicon Compounds. A. P. Kreshkov and V. A. Bork. *J. Anal. Chem.* (*U.S.S.R.*), **6**, 78–87 (March–April 1951).

Resins, paints, oils, and other technical products are examined for the presence of silicon by heating with sodium carbonate and sodium peroxide in a platinum loop and then testing the melt for silicate. Tests have been worked out for various important monomeric organosilicon compounds— Alkyl- and aryl-chlorosilanes react with an aniline soln. of tetramethyldiaminobenzophenone to give a characteristic reddish-orange ppt. Many organosilicon compounds, viz. tetraethylsilane, alkyl- and aryl-alkoxysilanes, and organosilicon compounds containing a phenyl group, give colorations or coloured precipitates when treated first with $AlCl_3$ and then with a benzene or aniline soln. of tetramethyldiaminobenzophenone; these reactions are described and tabulated. Tetra-alkylsilanes, unlike the other compounds examined, rapidly decolorise bromine on warming. Tetraphenylsilane gives silicic acid on warming with conc. H_2SO_4. A. E. S.

Standardisation of Dyes. G. H. Schuler. *Amer. Dyestuff Rep.*, **40**, P 378–P 380 (11th June 1951).

Problems of dye standardisation are discussed together with a description of methods and equipment. J. W. B.

Industrial Determination of Naphthols and Diazo Bases. F. Gasser. *Öster. Chem.-Ztg.*, **51**, 206–207 (1950): *Chem. Abs.*, **45**, 4176 (25th May 1951).

Diazo bases and KI interact with evolution of N_2 according to the equation—

$$R{\cdot}N_2Cl + KI = KCl + RI + N_2$$

By using special apparatus to measure the volume of liberated N_2 the amount of diazo base present can be calculated. Naphthols are first coupled with excess of a diazo base, and the excess of base is then determined. Neither method is very precise, but suffices for works control and comparison purposes. C. O. C.

Relation between Fibre Properties and Apparent Abrasion Resistance. D. D. Gagliardi and C. C. Nuessle. *Amer. Dyestuff Rep.*, **40**, P 409–P 415 (25th June 1951).

Accelerated abrasion tests show that the apparent abrasion resistance of fabrics modified by stabilising and creaseproofing compounds is greatly lowered by chemical treatments, the extent of lowering being directly proportional to reduction in fibre extensibility. An erroneous picture of fabric wearing properties is, however, presented by accelerated tests, since the fabric is subjected to high repeated cyclic stresses absent in normal wear. The rôle of improved fibre elastic recovery is not brought out by these tests. By using the Schiefer abrasion machine at decreasing applied loads, a point is reached where the modified fabric has a higher abrasion resistance by virtue of higher elastic recovery. Although no correlation was found between practical wear tests and accelerated tests, results support the contention that low-load abrasion resistance is more important for predicting wearing qualities. Stress is laid on the importance of wet abrasion resistance, which is increased by stabilising compounds applied to rayon fabrics. Introduction of short cross-links in cellulose produces the greatest reduction in apparent abrasion resistance, because fibre extensibility is reduced more than when long and flexible cross-links are introduced, in which case resistance is more easily retained. J. W. B.

Spectrophotometric Determination of Carboxyl in Cellulose. F. H. Forziati, J. W. Rowen, and E. K. Plyler. *Bur. Stand. J. Res.*, **46**, 288–291 (April 1951)— *Research Paper* 2197.

Carboxyl groups in oxidised cellulose can be estimated quantitatively from their infra-red absorption spectra. The estimation is made by preparing a mineral-oil slurry of a 1 : 1 mixture of polystyrene and cellulose, ground to a fine powder in a vibratory ball-mill, and comparing the C=O absorption band of the carboxyl groups at 5·8 μ. with the absorption band of the polystyrene at 6·2 μ., which is used as an internal standard. The method has been tested by comparing the results from cellulose oxidised in a variety of ways and estimated by the calcium acetate method. It was found that non-carboxyl carbonyl groups do not interfere. The method can detect as little as 0·1 millimole of carboxyl per gram. W. J. M.

Accessibility of Cellulose by Formic Acid Esterification. R. F. Nickerson. *Text. Research J.*, **21**, 195–202 (April 1951).

A method of estimating the structure of cotton by esterification with formic acid is described. It is simply carried out, and its advantage over the hydrolysis method is that there is no breaking of primary bonds and less possibility of recrystallisation. It is shown that negligible degradation of cotton occurs. The degree of accessibility of the cellulose structure as measured chemically is much more important than the actual degree of crystallinity as measured by physical methods, but the accessibility must be described in terms of the medium, whose swelling power will determine the fraction of the amorphous zone that is accessible. The cellulose structure may be divided into three distinct parts— (*a*) immediately available to e.g. water, (*b*) potentially accessible, e.g. on severe treatment such as beating, and (*c*) completely inaccessible. W. J. M.

Technique of Measuring Orientation in Cotton by X-Rays. R. Meredith. *J. Textile Inst.*, **42**, T 275–T 290 (July–Aug. 1951).

Crystallite orientation is measured for fourteen cottons by means of X-rays and is expressed (*a*) by the angular displacement from the point of maximum blackening on the (002) diffraction arc to the point where the absorption is 40% of the maximum, (*b*) by means of a calibration strip on each film which allows transmitted intensity readings to be converted directly into corresponding X-ray intensities, and (*c*) by taking into account the distribution of X-ray intensity around the (002) arc to give the average angle of inclination to the fibre axis of the chain molecules in crystalline regions. Good agreement is obtained between (*a*) and (*b*), which correlate less well with (*c*). J. W. B.

Relation between the Viscosity and Concentration of Cellulose Solutions in Cuprammonium; and the Calculation of the Intrinsic Viscosity of Cellulose. M. A. Calvert and D. A. Clibbens. *J. Textile Inst.*, **42**, T 211–T 234 (June); T 334 (July–Aug. 1951).

New experimental data are presented on the relation between the viscosity and concentration of solutions of cellulose in cuprammonium, particular attention being paid to the range of relative viscosities 1·5–10. Scoured cottons modified to different extents by acidic or oxidative degradation are chiefly used, covering an approximately

tenfold range of intrinsic viscosities. Several equations proposed to represent the dependence of viscosity on concentration are examined, and while satisfying the data in certain ranges of relative viscosity, they are not equally valid for the calculation of intrinsic viscosity. In the Baker equation an exponent of approx. 3·5 is required instead of the more usual 8, and in Farrow and Neale's equation a numerical constant much lower than the original 11 is suggested. The equation of de Jong, Kruyt, and Leno fits the data well over a range of relative viscosity up to 100, while Martin's modification represents the data fairly well with a single constant of 0·14. An approximate equation is suggested to facilitate the calculation of fluidities at a standard concentration of the solute from experimental measurements made at a slightly different concentration. J. W. B.

Wool Medullameter. R. E. Belin and C. L. Goldstone. *J. Textile Inst.*, **42**, T 332–T 334 (July–Aug. 1951).

A compact and sensitive instrument is described for measuring medullation in wool photoelectrically. Even illumination and good reproducibility are combined with rapid and easy operation. J. W. B.

Separation of α-Amino Acids on Powdered Cellulose Columns. S. Blackburn. *Chem. and Ind.*, 294–295 (14th April 1951).

Free α-amino acids may be separated on columns of powdered cellulose. Using 80% (vol./vol.) propanol–water as solvent, the addition of acid retards the basic amino acids and accelerates the acidic amino acids with respect to the neutral amino acids, whilst the addition of ammonia has the reverse effect. Thus a protein hydrolysate may be separated without the use of ionophoresis techniques by running on a cellulose column in 80% propanol made to 0·1 N. with HCl. The neutral and acidic amino acids run out fairly fast together. These are separated on a second column using 80% propanol made to 0·25 N. with ammonia. The fractions may then be further identified by running one-dimensional paper chromatograms in suitable solvents. Cellulose powder, however, is not as efficient as starch in separating amino acids. W. K. R.

Identification of *N*-2:4-Dinitrophenylamino Acids. A. G. Lowther. *Nature*, **167**, 767–768 (12th May 1951).

Although the dinitrophenyl derivatives of the longer-chain amino acids may be hydrolysed by heating with saturated baryta, satisfactory results are not obtained with the derivatives of the short-chain acids. It has now been found that short- and long-chain amino acids may be regenerated from their dinitrophenyl derivatives by heating with ammonia (sp. gr. 0·880) in a sealed tube for 2 hr. at 100°C. The free acids are liberated and then identified by paper chromatography. Under these conditions, cystine is decomposed and there is slight decomposition with proline and tryptophan, whilst dinitrophenylarginine does not react. Using this technique for investigation of the terminal amino groups of wool, leucine was detected in addition to the amino acids previously reported. W. K. R.

Assessment of Scouring Efficiency. M. Ringeissen. *Teintex*, **16**, 219–226 (May 1951).

The efficiency of a scouring process can be checked by extracting the scoured material with an organic solvent or by optical comparison with white material. A new method is described in which the material under test is impregnated uniformly with a fatty body coloured with a fat-soluble dye. The treated material is placed in the solution of the scouring agent under examination for a fixed time and at a standard temp., washed off, and compared colorimetrically with the original. The fatty body used should have zero acid value, and its iodine value should approach zero as far as possible. Palm oil and cooking lard are suitable products. The dye should be completely soluble in the fat used, insoluble in sodium carbonate and other alkalis, insensitive to reduction, homogeneous, and fast to light. B. K.

Determination of Sulphide Ion in Lime and Bate Liquors. R. Stubbings. *J. Amer. Leather Chem. Assocn.*, **46**, 297–300 (May 1951).

A quantitative colorimetric method for the determination of sulphide is based on the formation of a bluish-purple coloration by the reaction of sulphide ions with sodium nitroprusside. The colour is stabilised and changed to red-purple by the addition of guanidine. To 5 ml. sulphide solution (0·001–0·006 N.) are added 5 ml. guanidine buffer (10 g. guanidine carbonate and 6 g. NaOH per 100 ml.) and 3 ml. 1% sodium nitroprusside, and the volume is made up to 50 ml. After 10 min. the colour intensity is measured using a green (540) filter, and the sulphide content calculated by reference to a curve prepared from known standards. Lime and bate liquors are first freed from turbidity by filtration. Results are within ± 4% of those obtained by the iodometric method. W. K. R.

PATENTS

Colour Predictor for Pigments. American Cyanamid Co. and E. I. Stearns, Jr. *U.S.P.* 2,540,798.

There are two characteristics of pigment reflection, each being independently additive. One factor is the specific light absorption, which is practically the same as K, a function of body reflectance and dye concentration. The other is an analogous function for the light-scattering effect of pigment particles which, while not the same as, and changing in a manner quite different from, K, is nevertheless additive. The total light reflectance is proportional not to the sum but to the ratio of these two functions. Apparatus which uses this property of pigments to produce a quantity proportional to the overall reflectance of a mixture of pigments and/or substrates is described. C. O. C.

Determining by Continuous Integration the Tristimulus Values of a Mixture of Dyes. American Cyanamid Co. and R. H. Park. *U.S.P.* 2,542,564.

Measuring the Brightness of Light. General Electric Co., H. M. Archer, and H. A. Boltz. *U.S.P.* 2,542,299.

Testing the Fastness of Dyed Textiles to Washing or Milling. J. R. Geigy A.-G. *B.P.* 653,657.

Holders for the testing liquor fit into a heating bath. In each of these holders there is a reciprocable rubbing device which exerts regulable pressure on the pattern under test, which is placed between the rubbing device and the bottom of the liquor holder. C. O. C.

Testing and Recording Alterations in the Length of Yarn under Physical or Chemical Treatment. Celanese Corpn. of America and J. B. Steiding. *U.S.P.* 2,537,170.

Fabric Thickness Gauge. Goodall-Sanford Inc. and C. L. Bodwell. *U.S.P.* 2,538,858.

Measuring Heat-insulating Capacity of Textile Fabrics. U. Giandomenici. *B.P.* 654,534.

Fabric is supported on a frame so as to form a hermetically sealed chamber containing one or more adjustable sources of heat, these being regulated to maintain constant temperature inside. By balancing energy consumption against loss through the fabric walls, the heat-insulating capacity is determined. J. W. B.

Factors affecting the Service Life of Leather in Wool Processing. R. M. Koppenhoefer. (I, p. 379.)

Fractionation of Starch. V— Determination of the Molecular Weights of the Triacetates of Amyloses and Amylose Fractions by Osmotic Pressure Measurements. R. S. Higginbotham. (III, p. 380.)

Cotton Fibre Tensile Strength and X-Ray Orientation. R. Meredith. (VI, p. 392.)

Mechanism of the Hydrolytic Cleavage of the Inter-unit Bonds in the Cellulose Molecule. G. Beall and L. Jörgensen. (XI, p. 396.)

XV— MISCELLANEOUS

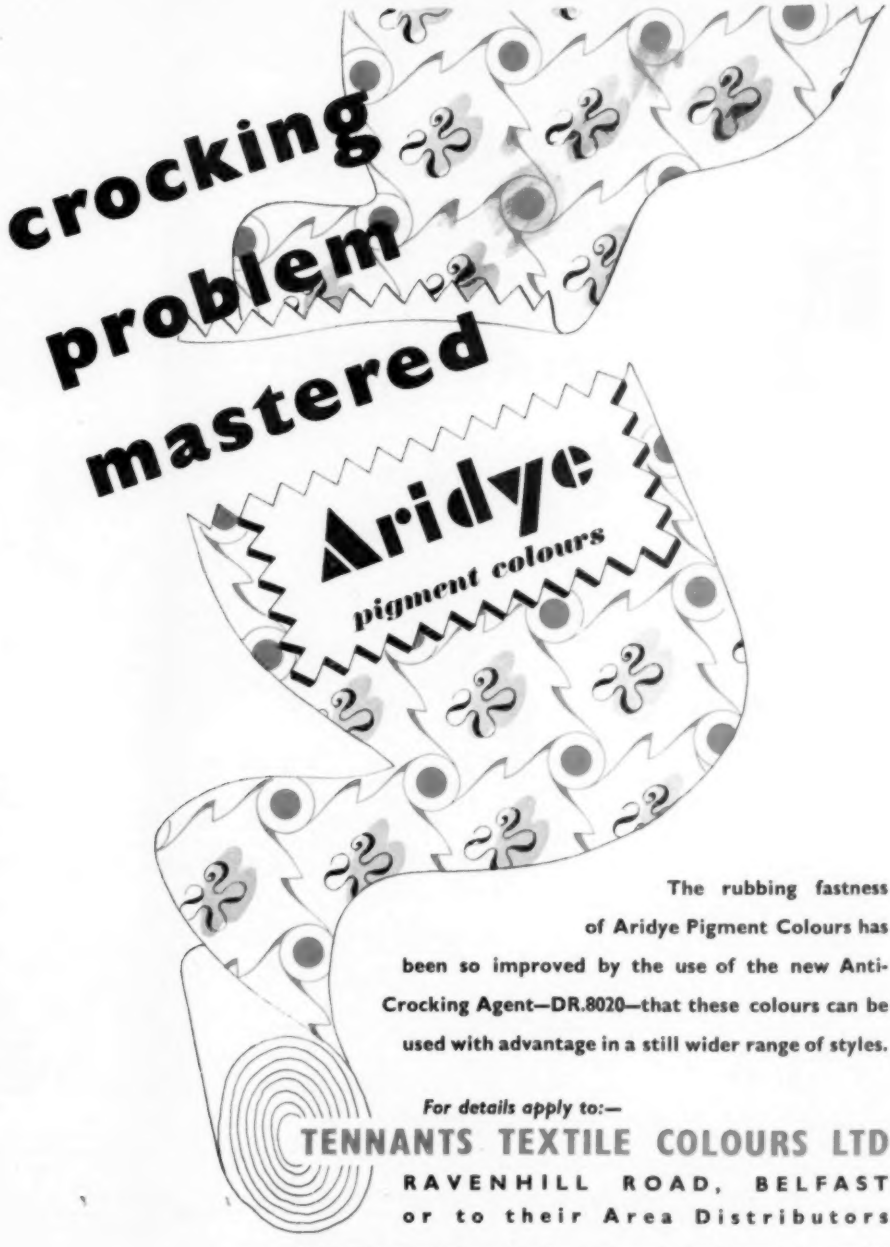
Symbols, Signs, and Abbreviations recommended for British Scientific Publications. *J.C.S.*, 1677–1692 (June 1951).

A report by the Symbols Committee of the Royal Society, representing also the Chemical, Faraday, and Physical Societies. C. J. W. H.

PATENT

Crayon for Marking Textiles. Uxbridge Worsted Co. Inc. *B.P.* 653,841.

A crayon, which marks cloth without glazing and setting into it and which is readily removed during wet processing, consists of clay and/or fullers' earth and/or infusorial earth and/or bentonite, a phosphate, and a colouring material. C. O. C.



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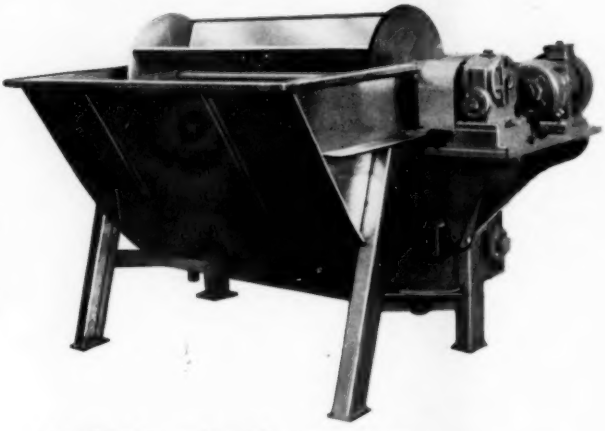

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FORTHCOMING MEETINGS OF THE SOCIETY—*continued*

NORTHERN IRELAND SECTION

All meetings to be held in Queen's Hotel, Belfast at 7.30 p.m.

1951

Wednesday 14th Nov.	C. P. Tattersfield, Esq. (Courtaulds Ltd.) *Some Dyeing and Finishing Problems with New Fabrics*
Wednesday 12th Dec.	R. W. Speke, Esq. (Imperial Chemical Industries Ltd.) *Continuous Dyeing with Vat Dyes*

1952

Wednesday 9th Jan.	Dr. G. H. Lister (Sandoz Products Ltd.) *Wool Dyeing*
Wednesday 13th Feb.	DISCUSSION EVENING
Thursday 6th March	R. C. McKinney, Esq. (Lilliput Laundry and Dyeworks Ltd.) *Commercial Laundering in Practice* (Joint Meeting with Textile Institute)
March	ANNUAL GENERAL MEETING and DINNER (date will be announced later)

SCOTTISH SECTION

All meetings at 7 p.m., St. Enoch Hotel, Glasgow

1951

Tuesday 30th Oct.	J. G. Evans, Esq., M.Sc.Tech. *Some Aspects of Synthetic Fibres*
Tuesday 13th Nov.	Professor J. B. Speakman, D.Sc., F.R.I.C., F.T.I. Details later
Tuesday 11th Dec.	J. Boulton, Esq., M.Sc.Tech., F.R.I.C., F.T.I. *Rayon Staple: A Dyeing Miscellany*

BRADFORD JUNIOR BRANCH

All meetings held in the Bradford Technical College at 7.15 p.m.

1951

Wednesday 31st Oct.	Visit to Courtaulds Limited, Westcroft Mill, Bradford
Thursday 15th Nov.	FILM EVENING. (1) *Not Just Velvet* (Lister & Co. Ltd.) (further details later)
Monday 10th Dec.	Dr. G. H. Lister (Sandoz Products Ltd.) *Textile Auxiliaries in Relation to the Textile Industry*

1952

Tuesday 22nd Jan.	STUDENTS' ANNUAL COMPETITION EVENING
Monday 18th Feb.	Dr. C. S. Whewell (Leeds University) *The Wet Finishing of Wool Textiles*
Thursday 28th Feb.	THE JUNIOR BRANCH DANCE at the Queen's Hall
Wednesday 5th March	J. A. Hepworth, Esq., B.Sc., A.T.I. (Shell Chemicals Ltd.) *Petroleum Derived Chemicals in the Textile Industry*
Saturday 8th March	Visit to Wool Industries Research Association, Torridon
Tuesday 25th March	E. E. Tallis, Esq., B.Sc., A.R.I.C. (Courtaulds Ltd.) *Recent Developments in Viscose Rayon and Staple Manufacture*

There will be a visit to Salts (Saltaire) Ltd., the date of which has not yet been fixed.

LEEDS JUNIOR BRANCH

All meetings to be held in the Colour Chemistry Lecture Theatre, Leeds University, at 4 p.m.

1951

Tuesday 30th Oct.	J. A. Hepworth, Esq., B.Sc. A.T.I. (Shell Chemicals Ltd.) *Petroleum derived Chemicals in the Textile Industry*
Tuesday 27th Nov.	Dr. C. H. Giles (The Royal Technical College, Glasgow). *Colour Photography*

1952

Tuesday 22nd Jan.	E. Stead, Esq., B.Sc. and A. Murray, Esq., B.Sc. (Yorkshire Dyeware & Chemical Co. Ltd.) *Some developments in Dispersed Azo Dyes for Acetate Rayon and Nylon*
Tuesday 5th Feb.	B. Kramrish, Esq., or J. A. Potter, Esq. (The Clayton Aniline Co. Ltd.) *The Dyeing of Vinyl Polymers*
Tuesday 26th Feb.	Dr. F. L. Warburton (Wool Industries Research Association) *Colour Vision*

MANCHESTER JUNIOR BRANCH

All meetings commence at 6.30 p.m.

1951

Friday 26th Oct.	Two papers by (*a*) Dr. W. Shaw of the Calico Printers' Association Ltd.; (*b*) Mr. Sutton of T. E. Marchington & Co. Ltd. *Cellulose Acetate and Vat Dyes.* Reynold's Hall, College of Technology, Manchester
Wednesday 7th Nov.	Visit to Standfast Ltd., Lancaster. For details contact the Secretary, Manchester Junior Branch
Friday 16th Nov.	Two short papers by post graduate research students of the College of Technology. Titles and lecturers to be given later. Gas Showrooms, Manchester

1952

Friday 11th Jan.	S. Burgess, Esq., H. Hampson, Esq., and E. R. Lambie, Esq., *The Application of Vat Dyestuffs to Viscose Rayon Packages.* Reynold's Hall, College of Technology, Manchester
Friday 14th Mar.	Lecturer from the Bleachers' Association Ltd. Name later. *Progress in Textile Bleaching.* Reynold's Hall, College of Technology, Manchester

WEST RIDING SECTION

All meetings held at the Great Northern Victoria Hotel, Bradford, at 7.15 p.m. unless otherwise stated

1951

Thursday 8th Nov.	Dr. F. Happey (Head of Dept. of Textile Industries, Technical College, Bradford). *Synthetic and Natural Polypeptides*
Thursday 22nd Nov.	C. C. Wilcock, Esq., F.T.I., A.R.T.C., and R. A. McFarlane, Esq. (Courtaulds Ltd.) *Some Comments on the Winch Dyeing of Rayon Fabrics*
Tuesday 27th Nov.	Professor W. Bradley. Title later. Metropole Hotel. Leeds
Thursday 6th Dec.	Dr. G. T. Douglas (I.C.I. Ltd.). *The Dyeing of 'Ardil' and 'Ardil' Cellulose Unions*

1952

Friday 18th Jan.	LADIES' EVENING

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Editors

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The Textile Institute and the Society of Dyers and Colourists have jointly published a Review of Textile Progress, Volume 1 relating particularly to the year 1949. There are 24 contributors in the Review and the authors are experts in the various sections for which they are responsible

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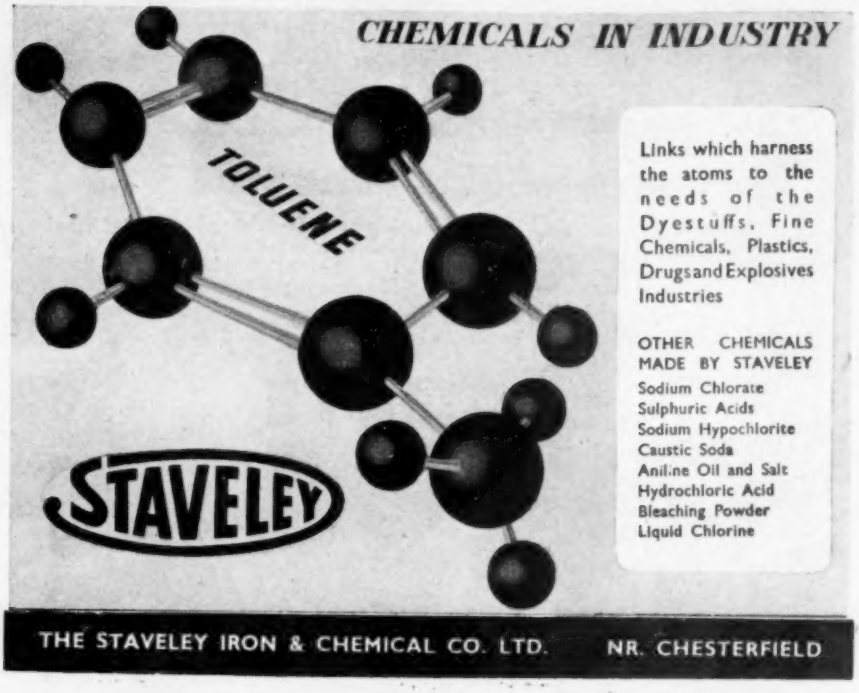

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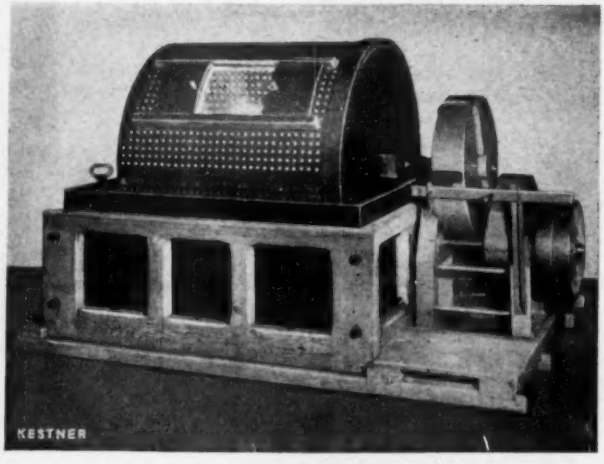

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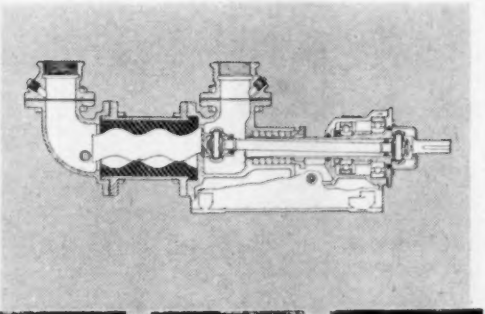

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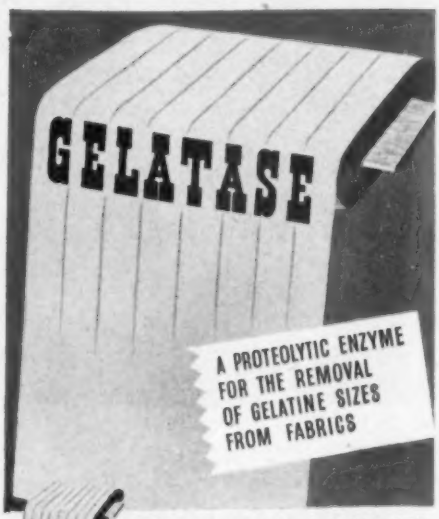

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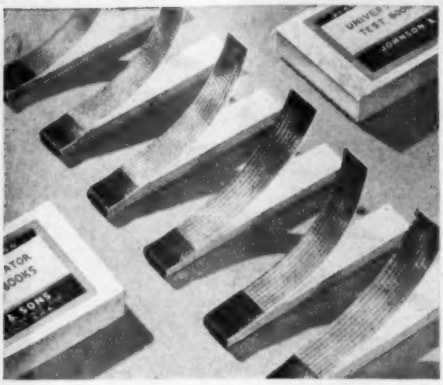

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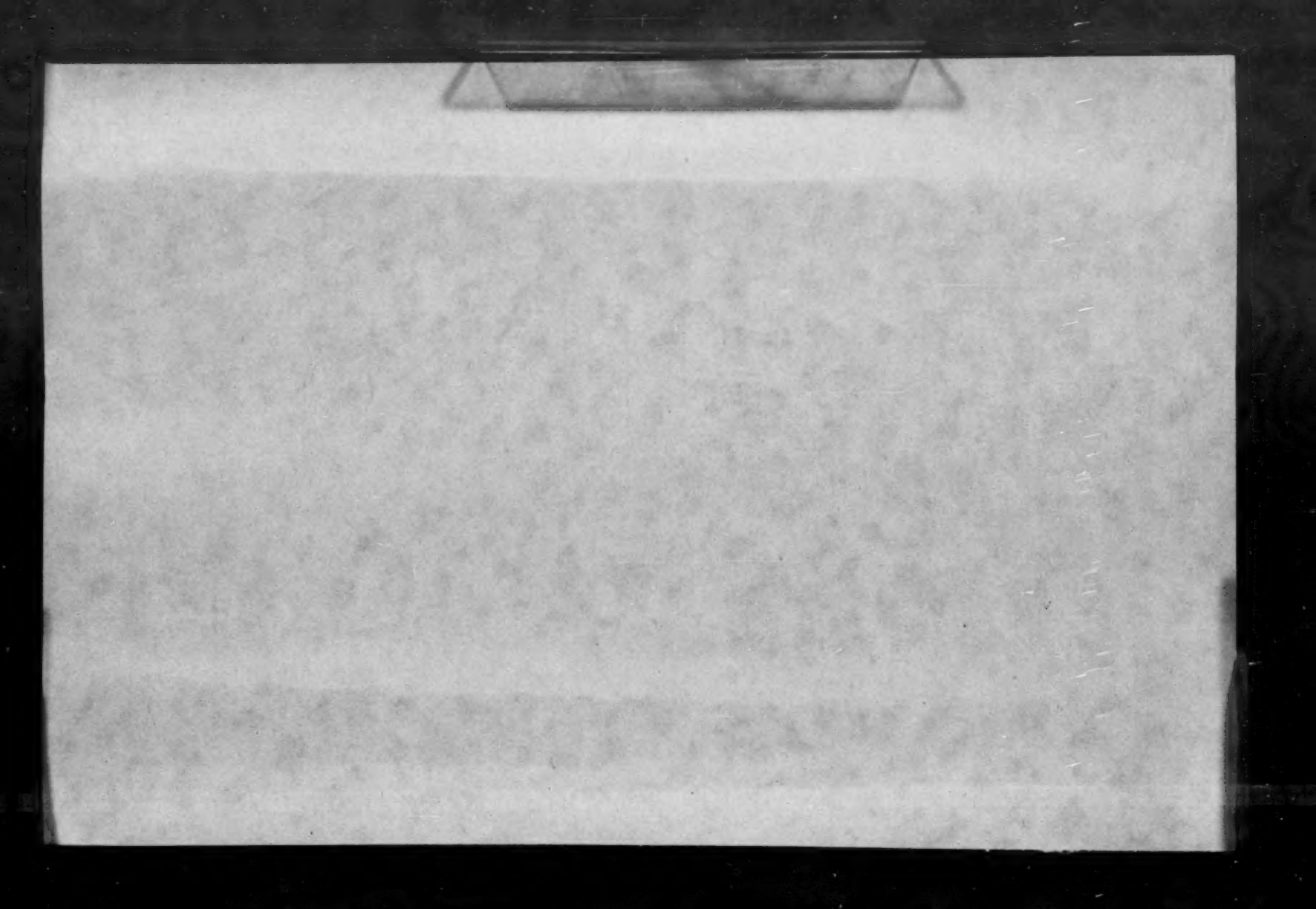

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